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Laboratory Hydrotransport Extraction System for Oil Sand Processing

By

Vincent Jay Wallwork ©

A thesis submitted to the Faculty of Graduate Studies and Research

in partial fulfilment of the requirements for the degree of

Master of Science in Chemical Engineering

Department of Chemical and Materials Engineering

Edmonton, Alberta

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Dr. Anthony Yeung
(Chair of committee)

Date: October 7/2003

Abstract

A modern laboratory scale apparatus has been developed to test oil sand. This apparatus was used to assess the extraction performance of oil sands under conditions analogous to current industrial processes. The apparatus has the capability of investigating independently, the liberation of bitumen from the sand as well as air-bitumen attachment and bitumen recovery. Experiments show that lower processing temperatures have a negative effect on bitumen recovery and that air addition has a positive effect on recovery. Air addition was shown to be more beneficial to poor processing ores and benefit from air can be achieved with a small air to slurry ratio. Additionally, liberation of bitumen from sand grains happens faster than air attachment and recovery, making the flotation the rate-limiting step. In an effort to improve bitumen-air attachment a novel use of hydrocarbon aerosol showed that flotation rate and recovery of bitumen can be markedly improved.

This thesis is dedicated to my parents, Margery and David Wallwork, for teaching me that I can do anything I put my mind to and providing an example for me to follow.

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Chapter 1 - Introduction

Bitumen reserves located in northern Alberta that are recoverable via surface mining techniques are estimated at 152 billion barrels (Alberta Energy and Utilities Board, 1996). Open pit mines operated by Suncor Energy Inc. and Syncrude Canada Ltd in the Fort McMurray region produce in excess of 400,000 barrels of bitumen per day. By 2010, expansion of existing plants and new construction are anticipated to increase crude oil production from oil sand to more than 50 percent of Canada's total production. Flotation of bitumen is the primary extraction technology applied to oil sands. Temperature, chemistry, surfactants, mechanical energy and composition have all been identified as contributing factors to processing characteristics. (Clark, 1944; Clark 1949; Sanford and Seyer, 1979; Bichard, 1987; Sanford 1983; Schramm et al., 1984 and 1985; Kasongo et al, 2000) This study engages the topic of air addition, in terms of quantity and delivery method, ore characteristics, and temperature on bitumen recovery. To discern the effects of these variables it is necessary to use a well-controlled experimental system. In such a system, the effect of each known variable, individually or in combination on bitumen recovery can be isolated and their critical level be identified.

A diverse and generally undefined terminology exists in literature discussing the oil sand industry. For clarity, the following terms will be defined here and used throughout this thesis. *Liberation* is the process of separating bitumen

from the sand matrix, involving ablation and detachment steps. Oil sand lumps, digested in either a tumbler or a hydrotransport pipeline, are subject to shear in the presence of added water and process aids. The ablation of oil sand is physically dependent on the temperature and shear forces present. Increasing temperatures greatly decreases bitumen viscosity, which along with high shear forces facilitates oil sand breakdown. The shearing action, coupled with the heating of the outer layer of an oil sand lump, ablates away the outer layer of the lump; thereby exposing fresh surfaces to be further heated and sheared away (Figure 1-1A). The sheared layers contain sand grains and/or fine clays covered with bitumen (Figures 1-1B). The detachment step consists of bitumen thinning (Figure 1-1D) that leads to pinhole formation (i.e., "pinning") shown in Figure 1-1E. The hydrophilic nature of sand grains, coupled with interfacial forces at bitumen/water interface causes bitumen to recess, so that distinct bitumen droplets are formed on the sand grains (Figure 1-1F). The liberation process illustrated by steps from D to F in Figures 1 is followed by bitumen detachment from the sand grains. As well, bitumen within the sand pores becomes liberated due to shear action in the hydrotransport pipeline. The second process is *recovery*, which includes air attachment and gravity separation steps. The bitumen droplets attach to air bubbles (air attachment step, Figures 1-1G and 1-1H) based on physical and surface properties of the air bubbles and bitumen droplets. There is no reason to believe that the aforementioned steps occur sequentially and it is quite likely that bitumen

liberation from the outer layer of an oil sand lump takes place simultaneously with the already sheared bitumen-sand layers. As well, air attachment can take place prior to and facilitate liberation as long as bitumen and air are available to each other.

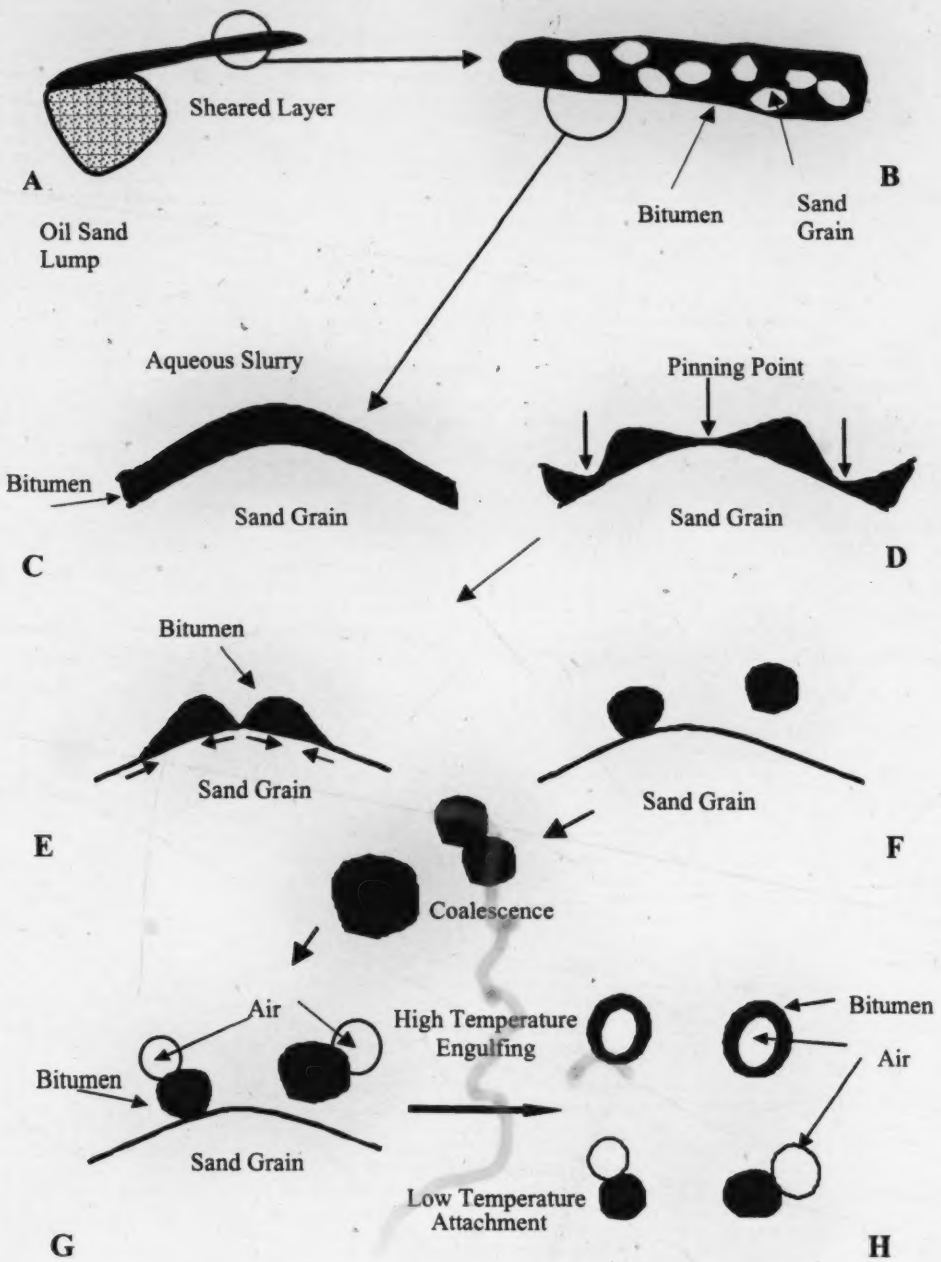

Figure 1-1 Conceptual stages for bitumen liberation and aeration (Masliyah, 2002)

Different technologies have been evolved in commercial oil sands processing, partly due to the advancement of our understanding on oil sands processability and partly due to the environmental restraints. The Clark Hot Water Extraction process (CHWE) (Hepler and Smith, 1994), employed by both Syncrude and Suncor was the first commercial process applied to the oil sands. In this process, bitumen liberation and aeration occur in a tumbler by mixing oil sands with water, steam and often a small amount of caustic at a temperature above 75°C. The conditioned slurry is then subjected to gravity separation to recover the aerated bitumen. In the 1990's, the incorporation of slurry transport of the mined oil sand using pipelines led to a departure from the CHWE process to one where the conditioning and aeration of the oil sand occur in a pipeline. With the use of a pipeline, the tumbler has been eliminated and the conditioned slurry from the pipeline is delivered directly to gravity separation vessels. The operating temperature has been reduced to approximately 50°C in what has become known as the Warm Water Process (WWP). Recently, the construction of satellite oil sand projects such as Syncrude's Aurora mine coincided with the introduction of the Low Energy Extraction (LEE) process. This process and others such as the OSLO Cold Water Extraction (OCWE) process (Sury, 1990) are hydrotransport based processes that operate at temperatures less than 35°C. This continues a trend of separating bitumen from oil sand at lower temperatures.

With the push for lower processing temperatures by industry experimental research into the effects of lower temperature on oil sand processability was a natural direction to take. Chapter 2 contains a brief literature review on different laboratory apparatus for processability tests of oil sand. Several different constructions have been developed and used over the years, with the most successful being Syncrudes Batch Extraction Unit. As well the role of temperature and air in bitumen extraction as seen in literature are presented.

This thesis focuses on the development of a new and novel laboratory scale test facility suitable for examining temperature and air addition in the context of bitumen extraction from oil sand.

Initially, tests at lower temperatures (below 50°C) were carried out with a Syncrude Batch Extraction Unit. These experiments are described in Chapter 3. Several challenges were encountered while performing these experiments that were directly related to processing oil sand with this equipment at low temperatures. It was determined at the time that the BEU's shortcomings as a laboratory testing tool warranted the attempt to design and build a better Laboratory unit for testing processability of oil sand in low energy and temperature process.

Chapter 4 describes the design and construction of the new piece of equipment named the Laboratory Hydrotransport Extraction System (LHES). This equipment was designed to resolve some of the shortcomings of the

BEU with regard to low temperature operation as well as reflect more closely modern industrial processes.

The LHES was utilised after construction to continue investigating the role of temperature and aeration in oil sand extraction. Since aeration has received little attention in the literature and is of increasing importance as temperatures are lowered it was a logical area to study. Chapter 5 presents the experimental results and some discussions relating to the findings.

Use of the LHES revealed that air attachment was a fundamental weak link in bitumen extraction. Coincidentally work by others led to the idea of improving air-bitumen attachment through the use of "Oily Bubbles". Chapter 6 describes this advancement and some experiments that demonstrate its potential.

Finally Chapter 7 draws some conclusions from the work presented here followed by recommendations and suggestions for future work in Chapter 8.

Chapter 2 - Literature Review

2.1 Laboratory Oil Sand Testing Equipment

The oil sand industry has made considerable progress in terms of process development. However, laboratory testing methods for investigating oil sand processability are based on the original Clark hot water process. The original laboratory-scale separation unit which took an 8 Kg charge for each test, was developed by Clark to investigate oil sand processability during his development of the Clark Hot Water Extraction process (CHWE) (Clark, 1932; FTFC, 1995). A stirred reactor with a v-shaped paddle was developed later. With the proper manipulation of variables, the reactor using a 0.2Kg charge could simulate pilot-plant extractions (Sanford 1979). A variety of stirred vessels have been used at different times, including conventional flotation machines such as the Denver Cell, more recently oil sand conditioning tests were carried out by bench scale rotating tumblers (Grant 1977, Mckay 1978, Mckay 1980) and by plate mill (McKay 1981)

The principal workhorse of laboratory testing equipment for oil sand processability assessment over the last 30 years has been the Batch Extraction Unit (BEU) (Figure 2-1) developed at the Syncrude Research Centre (Sanford and Seyer, 1979). This unit was developed to mimic the trends found in the CHWE process as employed commercially by Syncrude. The unit used a 0.5 Kg sample and consisted of a square pot with a variable

speed stirrer that also sparged air into the pot. Much of the knowledge about the chemical and interfacial effect on the processability has been generated from thousands of experiments conducted with this unit.

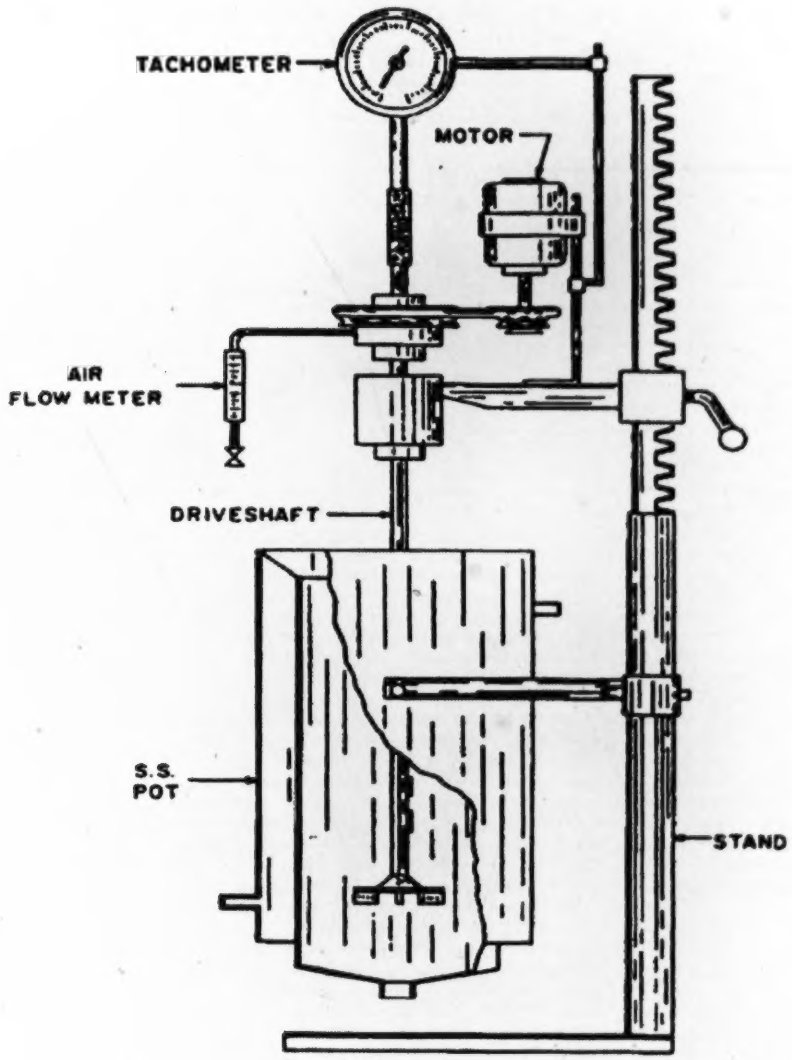

Figure 2-1 Laboratory scale batch extraction unit, BEU, for the study of bitumen extraction (Sanford and Seyer, 1979)

More recently, a modified bench-scale Denver flotation cell was employed to investigate the role of calcium ions in bitumen extraction (Kasongo, 2000). The development of low temperature processes has mostly been accomplished with small-scale pilot plant tests that are well suited for proving technology development and process design but less suited to fundamental investigation of oil sand processing characteristics.

2.2 Role of Temperature in Bitumen Extraction

Temperature has always been a principal variable in the extraction of oil sands. The general conclusion in literature is unanimous that higher temperatures are better for bitumen extraction.

Tests performed at the Great Canadian Oil Sands plant, the first commercial plant to operate using the Clark Hot Water process showed that good recovery of bitumen could be achieved at temperatures between 65°C and 80°C. Recovery dropped as the temperature dropped below 65°C (Innes, 1967). Sepulveda also reported that for oil sand ores found in Utah, USA, recovery dropped as the temperature decreased, with a significant falling off at temperatures below 60°C (Sepulveda, 1978). Reduced recovery at lower temperatures was attributed to increased bitumen viscosity and accompanying reduction in bitumen-air attachment and engulfment (Czarnecki, 1991; Dai, 1996). Another study comparing two different oil sands at 50°C and 15°C showed a markedly different effect: in one case increased temperature resulted in higher bitumen recovery but in the second

no difference was found. In the first case it was concluded that bitumen viscosity was the reason for the difference while in the second high fines content negated the effect of increased temperature. The conclusion in the second test was that it was the viscosity of the entire bulk *system* that determines the air-bitumen attachment efficiency (Lam, 1992).

Temperature also plays a role in the liberation of bitumen from sand grains. The decreased viscosity by increasing the temperature enhances the mobility of the bitumen and its potential to be sheared away from the sand grain. Additionally higher temperatures increase the repulsive interaction force and decrease the adhesive force between bitumen and silica therefore enhancing liberation of bitumen from sand (Liu, 2002)

2.3 Role of Air in Bitumen Extraction

Air is needed to reduce the apparent density of the bitumen such that it can be floated from the clean sand and water via gravity separation. Air can come from the ore itself, as dissolved air in the process water, become entrained in the slurrying process or be added as a discrete air stream to the ore slurry. The effectiveness of the air depends on its attachment to bitumen, which is a function of collision frequency, air bubble and bitumen droplet size, film thinning and rupture properties (induction time) and attachment strength (Yoon, 1989; Sury, 1993)

It has been shown that the best flotation performance occurs when air bubbles have the same or smaller size than the bitumen or mineral particles

that are being recovered. It is also reported that larger air bubbles and bitumen droplets are better than smaller ones (Takamura, 1988; Wu, 1999; Alexander, 1996). There are many aspects that impact the size of bitumen droplets and air bubbles. Bitumen content, sand grain shape and size, mechanical and thermal energies in the system, type and amount of fines and clays, water chemistry and pH all contribute.

Air-bitumen attachment and bitumen flotation are a function of hydrodynamic, surface chemical, colloidal, hydrophobic and thermodynamic interactions in a system (Takamura, 1988); Okada, 1990; Takamura, 1985; Zhou, 1998)

The induction time, or the time it takes while a bitumen droplet and air bubble are in contact for attachment to be made is strongly influenced by system temperature, water and particle surface chemistries, particle hydrophobicity and mechanical energy (Tyerman, 1995; Yoon, 1989; Gu, 2002; Gillespie, 1956)

Once attachment is achieved recovery efficiency depends on the hydrodynamic and rheological conditions in the separation vessel and the strength of bitumen attachment to the air. The attachment strength is a function of interfacial thermodynamics (Tyerman, 1995; Alexander, 1996; Drelich, 1995; Drelich, 1995b)

Chapter 3 - BEU Testing

Initially the Syncrude BEU was selected to investigate the role of temperature and air on the processability of different oil sands. The first experiments performed in this study were an investigation of the effect of temperature on bitumen recovery by standard BEU tests.

3.1 Experimental

3.1.1 Experimental Procedure

The experimental procedure adopted was modified from that outlined in Syncrude Analytical Methods for Oil Sand and Bitumen Processing (Bulmer, 1979). The detailed procedure is as follows.

A precisely weighed 500 g of oil sand sample was placed in a beaker. About 1500 ml of de-ionised water was heated to a desired temperature. The pot containing 150 grams of water was allowed to equilibrate with to the temperature of the circulation water. The pot was raised and impeller turned on to 600 rpm. The pre-weighed sample was transferred to the pot. The air was allowed to flow through the pot at 150 ml/min.

The sample was mixed in the pot for 10 minutes while moving the pot up and down a few times to break up any lumps of oil sand that may be present. After the mixing stage the airflow was stopped and 850 ml of water, preheated to the correct temperature, was added. The diluted sample was

then mixed for an additional 10 minutes. After 10 minutes had elapsed the mixer was stopped and any froth present was skimmed and transferred to a pre-weighed jar. This froth is referred to as *primary froth*. After the primary froth is collected the air was turned on to a rate of 50 ml/min and the sample mixed for 5 minutes at 780 rpm. While the sample is being mixed any froth that was generated was skimmed and collected in a second pre-weighed jar. This froth is referred to as *secondary froth*. After the secondary froth is collected the contents of the pot, referred to as *tailings,* were drained into a 1.5 litre jar. The final test step is to wash the pot, impeller and spatula used for froth skimming with toluene into a pre-weighed jar and denoted as *wall froth.*

3.2 Results and Discussion

Figure 3-1 shows that as the temperature decreased from 55°C to 25°C the recovery rate dropped. The recoveries shown are primary froth collected over time, with the final cumulative value after 15 minutes constituting the primary recovery as per standard BEU procedures. This result can be explained if one considers that the viscosity of the bitumen decreases with decreasing temperature. As the bitumen viscosity decreases the rate of liberation of the bitumen from the sand grain is slowed and the attachment strength between the bitumen and the air bubbles is reduced as the bitumen is less mobile and less able to engulf the air bubbles, providing weaker bitumen-air attachment.

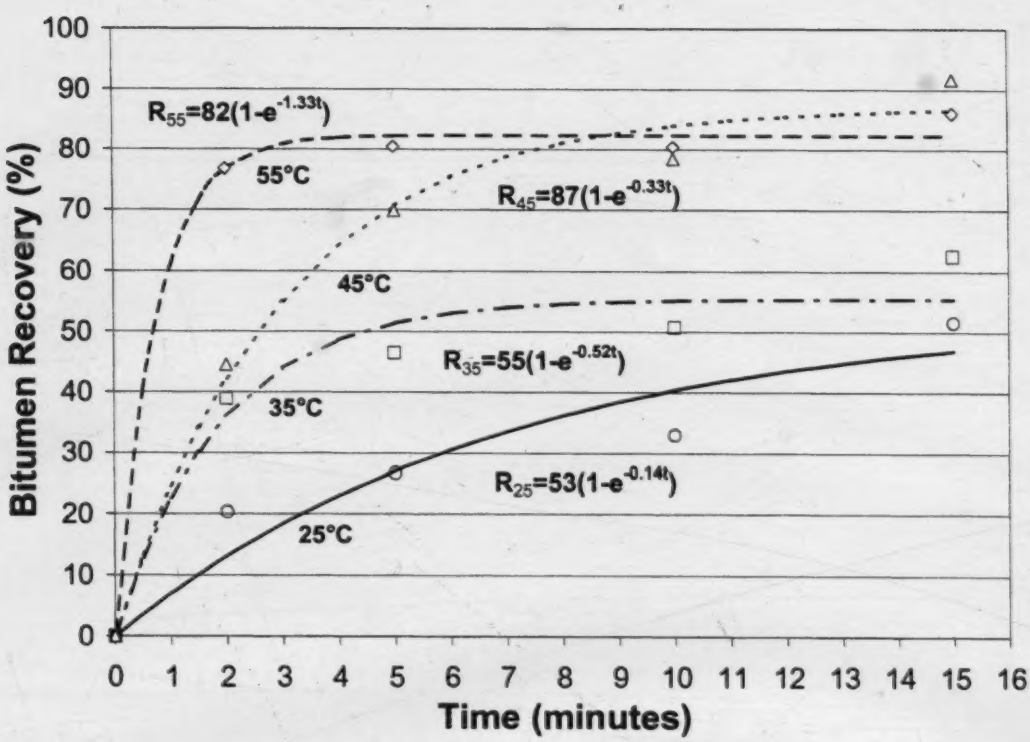

Figure 3-1 BEU tests using "Good" processing ore with 12% bitumen grade. The lines are data fit to the 1st order flotation recovery equation with the fitted parameters given by corresponding recovery equations.

It is evident in Figure 3-1 that the ultimate recovery for the tests at 45°C and 55°C is higher at 87 and 82 percent than the ultimate recovery for the 35°C and 45°C tests, with ultimate recoveries of 53 and 55 percent respectively. It is hypothesised that between these two temperatures is the transition from bitumen attached as a particle (lower temperature) to bitumen engulfed around the air bubble. The weaker attachment of bitumen particle-air bubble could result in lower flotation efficiency and lower bitumen recovery.

There were many difficulties that were encountered while testing oil sands in the BEU at temperatures lower than 50°C. Most notable is the recoveries being unrepeatable. Some observations that might account for this variability in results include oil sand getting lodged in the corners of the pot and not being mixed properly, substantial amount of oil sand hanging up on top of the impeller and excessive amounts of wall froth. In general it was found that the BEU did not perform consistently at lower temperatures with the procedures outlined for the unit.

Along with the issue of repeatability, concerns regarding difference in the conditioning forces in the BEU compared with the conditioning in a pipeline and the inability to measure the impact of liberation on the dynamic flotation rate of the liberated bitumen prompted the effort to design and build a new laboratory scale apparatus for better testing oil sand extraction.

Chapter 4 discusses the concept and design of a new laboratory scale extraction device for testing oil sand.

Chapter 4 - LHES Design and Construction

To meet the needs of development in the oil sands industry and to provide in-depth knowledge of oil sand processability, a laboratory scale set-up, which can approximate a variety of modern commercial extraction processes, is urgently required. For this reason, a Laboratory Hydrotransport Extraction System (LHES) was constructed. This is the first bench scale testing equipment that reflects modern oil sands industrial processes. At the same time, it offers new analytical advantages that will improve the understanding of oil sand processing characteristics.

4.1 Design Concept and Description

In designing a laboratory testing equipment, the issues of sample size and waste management must be balanced with the ability to get reliable data, thus the sample size is of concern. The sample must be sufficiently large so that bitumen losses on the wall of the unit are relatively small and that bitumen sample size is suitably large for accurate assaying. Additionally, the sample must not be so large that management of wastewater and tailings becomes prohibitive. For these reasons the LHES was designed using 17mm inside diameter heavy wall glass pipe with an outside diameter of 25mm. The use of glass stops bitumen from adhering to the pipe walls and the small size keeps the sample size manageable and the slurry velocities in the approximate range of industrial operations. The unit is approximately 3

meters in total pipe length and holds a total volume of 4 litres. A sample size ranging from 1 kg to 3 kg can be used depending on the slurry density that is desired. Oil sand testing is time consuming and thus extracting the maximum amount of information from each experiment is a priority. Application of visualisation techniques to the slurry and the aerated bubbles further generates the information that provides insights into the process from a single test.

The primary component of the LHES is the loading/flotation container (Figure 4-1A). This open top container allows addition of dry oil sand feed to the circulating water in the unit to form slurry. As the bitumen is liberated and aerated, the open slot allows the aerated bitumen to float to form a froth layer at the top of the container, which can then be collected. The container is wedge shaped so that turbulent eddies become weakened at the wider top of the container but remain strong at the narrow bottom. This allows a quiescent froth layer to form at the top while suspending the coarse solids at the bottom and at the same time maintaining a minimum container size. Incorporated at one end of this container is a narrow slot 25 mm wide and 3 mm thick (Figure 3B) (Zhou, 2000). This slot is open at the top and narrows to a 3mm square opening at the bottom. The purpose of this slot is to allow aerated bitumen and bubbles to enter from the bottom and to be viewed, and recorded through a macro lens system and CCD camera. A small counter current flow of clean water is introduced at the top of the slot to wash entrained slurry water out of the field of view. The addition of this counter

current flow ensures good image quality throughout the test. A spout opposite the visualisation slot allows collection of overflow froth. The slurry is transported in the loop with a low speed progressive cavity pump. Compared to a centrifugal pump the progressive cavity pump (Figure 4-1C) imparts very low shear forces on the slurry. The velocities in the pump are the same or less than that in the pipe section of the equipment. This design minimises the "conditioning" of the slurry by the pump. The pump is driven by a 3 Hp variable AC drive that allows precise speed control. Setting the speed of the positive displacement pump controls the flow rate. The pump is coupled to the glass pipe sections with flexible PTFE connectors, allowing flexibility between the rigid pump and the glass pipe, while reducing the stress on the glass. At the pump discharge, a lower drain valve (Figure 4-1D) is installed at the lowest point in the loop for the drainage of any slurry remaining at the end of the test. A port (Figure 4-1E) is installed to inject air through an 18-gauge needle in to the slurry stream. Next to the air injection port is a pressure relief valve (Figure 4-1F). This valve protects the glass tubing from being over pressured by the positive displacement pump in the event the line plugged. The bottom glass section includes a double pipe heat exchanger section that is connected to a programmable circulating bath. The circulating bath can either cool or heat the slurry as needed. The vertical end section is connected with glass ball and socket joints (Figure 4-1H) to allow flexibility and minimise stress on the sections of pipe. A second port is positioned at the top left of the loop (Figure 4-1I) for a thermocouple to

be inserted in to the slurry stream. The thermocouple is interfaced with the circulating bath to control the slurry temperature. Figure 4-1J shows the slurry visualisation camera. This camera, oriented horizontally to the pipe, takes images as the slurry passing through a squared section of glass. An accompanying PC captures the images and the grey scale information is analysed in real time. By analysing the relative darkness of the images, the degree of liberation of bitumen from the sand grains can be determined. A more detailed discussion of this technique will follow. Between the slurry visualisation camera and the loading vessel a set of ball valves (Figure 4-1K) are positioned to redirect the slurry stream. These valves are set at the end of the test to divert the slurry out of the loop into a collection container. Clean water is poured in the loading container to flush the slurry out of the system. Approximately 2 to 3 L of water is needed to clean the loop thoroughly. Any remaining water is then drained out of the system via the lower drain valve.

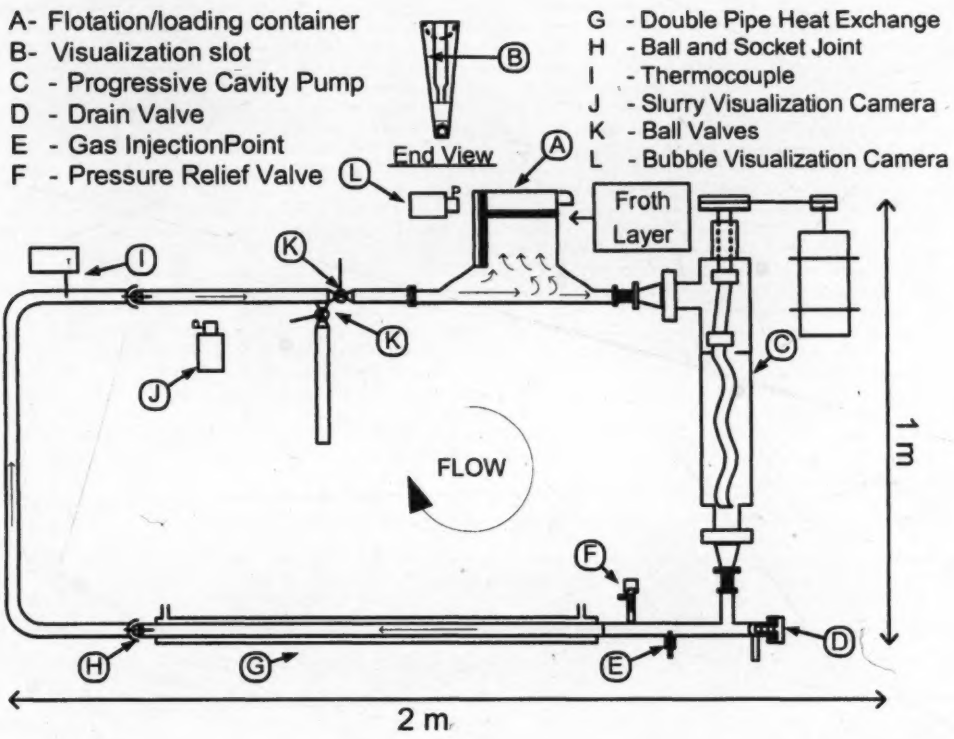

Figure 4-1 Schematic of Laboratory Hydrotransport Extraction System (LHES)

The open container (see Figure 4-1A) allows bitumen droplets that have been liberated and aerated to float and form a froth layer at the top of the container. The froth is collected at suitable time intervals in order to determine bitumen recovery rate. A small spatula is used to transfer the froth to the collection thimbles. Whatman filtration thimbles from Fisher Scientific are used as the collection containers so that the samples can be placed directly into modified Dean Stark extractors for froth composition analysis (Hepler, 1989 and 1994, Bulmer 1979).

4.2 Froth Composition Assays

The froth samples are separated into bitumen, water and solid components by refluxing with toluene in a modified Dean Stark extraction apparatus. Co-distilled water and toluene are condensed into a trap whereby the water is collected in a graduated section while the toluene is allowed to reflux through the extraction thimble. The filtered solids are dried and the resulting bitumen/solvent solution is centrifuged to separate non-filterable solids. An aliquot of the centrifuge supernatant is evaporated to remove the solvent. The mass of the water, solids and bitumen is determined gravimetrically.

4.3 Liberation Determination

The liberation process is the primary step in extracting bitumen from oil sands. Understanding the impact of processing conditions on the rate of bitumen liberation has not received much attention. Since liberation is a necessary step for bitumen flotation, the ability to measure the degree of liberation with time, concurrent with the flotation rate is advantageous. Luthra (2001) showed an alternative method for estimating bitumen recovery, based on visualisation of oil sand slurry undergoing digestion. Adaptation of his approach has led to the following method for measuring bitumen liberation based on the same principles of grey scale image analysis.

Since a bitumen covered sand grain is relatively dark and a clean sand grain or clay particle is relatively light coloured, it is possible to distinguish between

the two based on this contrast. Experience has shown that a bitumen coated sand grain has a grey scale value much less than 100, where 0 is black and 255 is white and that liberated sand grains have a grey scale value much greater than 100. For this reason a grey scale threshold of 100 can be comfortably chosen as sufficient to differentiate between bitumen coated or "black" sand grain and an uncoated sand grain. Tests done so far revealed that the liberated bitumen drops were effectively invisible to the camera, making the unrecovered, but liberated bitumen in the slurry insignificant to the liberation measurement. For example, the same oil sand, one with the bitumen recovered as it was liberated and a second where the bitumen was left, in a liberated state, in the slurry showed no difference in the measured liberation. To account for this observation it was hypothesised that the liberated bitumen droplets, being much smaller than the area measured by a single pixel, which in this arrangement was 25x25 μm, were essentially lost in the surrounding light coloured slurry.

The video images (Figure 4-2) acquired by the slurry visualisation camera (Figure 4-1J) are digitised by an accompanying PC into a 640 by 480 pixel resolution 8-bit greyscale bitmap image. These images are analysed using custom-programmed software that analyses the images in real time, 15 frames per second. The number of pixels that have a grey scale value below 100 are summed, providing an indication of the amount of bitumen-covered sand grains in the image. It was assumed that at the beginning of the test that all of the bitumen is associated with sand grains; therefore, the sum of

the black pixels throughout the test was divided by the number of black pixels at the beginning of the test. This ratio representing the fraction of bitumen that remained associated with sand grains, was subtracted from 1 to convert it to the fraction of bitumen liberated, where 1 indicates a complete liberation of bitumen, i.e. no bitumen is associated with sand grains. Mathematically, the degree of liberation is defined:

$$L\% = \frac{N_0 - N}{N_0} * 100 \qquad (1)$$

where N_0 is designated as the number of "black" pixels at time zero and N, the number of "black" pixels at a given time t. The approach adopted here was to average the values of L into one minute bins to smooth the results and reduce the data to a manageable size.

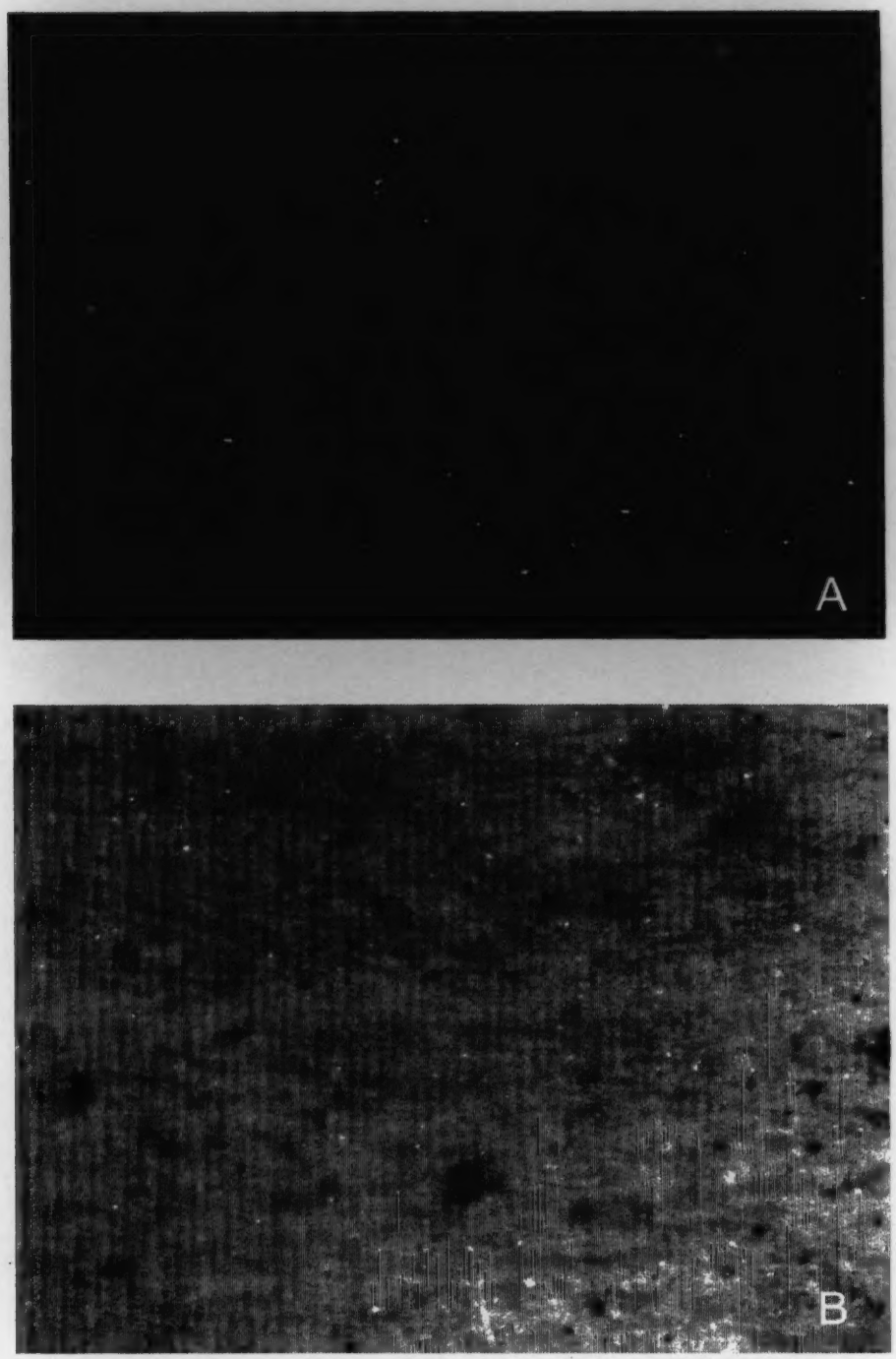

Figure 4-2 a) Slurry image at the start of a test. b) Slurry image after 60 minutes.

4.4 Standard Semi-Batch Mode Procedure

The majority of the tests using the LHES were performed using the following procedure. The term semi-batch was adopted since the slurry undergoes conditioning and flotation concurrently, rather than undergoing discreet process operations as it would in a true batch type process.

All valves were set and connections sealed before the test began. The circulating bath was allowed to reach the desired temperature for controlling the experiment depending on whether cooling or heating was required. Approximately 4 L of water was heated to approximately 20°C above the testing temperature. A suitable quantity of oil sand for the desired test or tests was homogenised and a 50 g sub-sample was retained for oil/water/solids analysis. The oil sand sample for the test was precisely weighed and any lumps larger than 1 cm crumbled by hand.

The preheated water was added to the LHES, care was taken to leave enough room to add the oil sand without overflowing the apparatus. The water in the loop was allowed to reach equilibrium before the oil sand was added as quickly as possible. After the oil sand was added the timer was started along with the slurry video analysis. The air rate was adjusted to the desired amount. Finally, any additional water needed to fill the system and bring the froth layer to the top of the vessel was added.

The unit was operated under consistent conditions for a desired time (usually one hour) while collecting the entire froth into specified intervals. The

intervals were spaced such that roughly equivalent amounts of froth are collected at each interval. The froth samples were analysed for oil/water/solids composition. At the end of the test the discharge ball valve was opened and the inline ball valve (valve K, Figure 4-1) was closed. The slurry was flushed out of the loop with approximately 2 – 3 litres of clean water.

After flushing the slurry out of the system, reset the ball valves and drain any remaining flush water out the bottom drain valve (Valve D, Figure 4-1). Close valve F and circulate exactly 1000 ml of toluene through the loop to dissolve any bitumen adhered to the system. Drain the toluene out valve D and reserve a sample for centrifugation and bitumen content via gravimetric analysis. Close valve D and circulate approximately 3 litres of soapy water to emulsify any remaining toluene and solids. Flush the soapy water out valve K with clean water, and finally drain the system via valve D.

4.5 Standard Full Batch Mode Procedure

This procedure was developed and tested but not used for any experiments in this thesis. The concept was to seal the pipe loop of the LHES such that the slurry can be conditioned much the same way as a commercial pipeline. Air added to the slurry could not escape in this arrangement. Once the slurry was conditioned it was flushed into a square glass cell and suspended with a mixer where aerated froth could float and be recovered.

A second vessel shown in Figure 4-13 was designed to seal the pipe loop when operating in batch mode configuration. The 50 mm diameter column atop the pipe was open for adding the slurrying materials. After loading, the column was sealed with a Teflon plunger. The plunger had a fine mesh screen and a drain port through it in order to allow excess water to escape when installing the plunger in the column.

If operating the unit in full batch mode the slurry was flushed from the system through the ball valve arrangement into static separation vessel. This square glass vessel contained the slurry and additional floodwater used to flush the slurry from the pipe loop. A fractional horsepower mixer is used to suspend the solids in the lower half of the vessel allowing aerated particles to float and be recovered.

The following procedure details operation of the LHES in this fashion. The desired slurry volumetric composition in terms of water, oil sand and air were determined. The water and oil sand prior to testing was precisely Measured out. The water was added to the system and brought to the desired temperature, by preheating if necessary. The system velocity was set and dry oil sand added though the column vessel and the plunger was install in the full down position using the plunger vent port to release air and any excess water in the column section of the vessel. The slurry video analysis and recording operations on the PC were started. If desired, additional air was introduced though the injection port, displacing water through the vent port in the plunger. The slurry was conditioned for the desired time. After

conditioning the discharge ball valve was opened and the inline ball valve closed, flushing the slurry into the square glass floatation box using flush water added thought the column. The flush was done with the desired amount of floodwater, pre-heated to the correct temperature. The mixer was activated to suspend the solids in the lower half of the floatation box and agitate the slurry until all aerated particles were floated and recovered. The froth was analysed for oil/water/solids composition.

Chapter 5 - LHES Experiments

5.1 Qualitative Observations

The bubble visualisation slot (Figure 4-1B, Figure A-12) allows detailed view of the nature of the bitumen attachment and amount of loading of the air bubbles in the system. Figure 5-1 gives a qualitative example of the difference between low fines content ore shown in a) and high fines ore shown in b) taken during a typical experiment. The ore was processed with de-ionised water at 50°C. It is evident that in a) the bubbles are heavily laden with bitumen while with high fines ore the bubble only has a spotty film of bitumen. It is interesting to note that at this temperature the bitumen attachment is of a particle-bubble type as opposed to engulfment observed for operations at CHWE process. Some other visual observations were made regarding the froth quality. In general, froth color darkened as the test progressed. This could be an indication that fine clays with a propensity to attach to air are floated at the beginning of the test giving the froth a lighter, dirtier appearance. Other data from unrelated tests using the LHES showed that the particle size distribution of the froth at the beginning of the test was very close to that of the feed. This would suggest that a very effective flotation system was in play and the lighter color could be due to the size distribution of the solids and the likelihood that the bitumen-mineral aggregates were being floated rather than fully liberated bitumen.

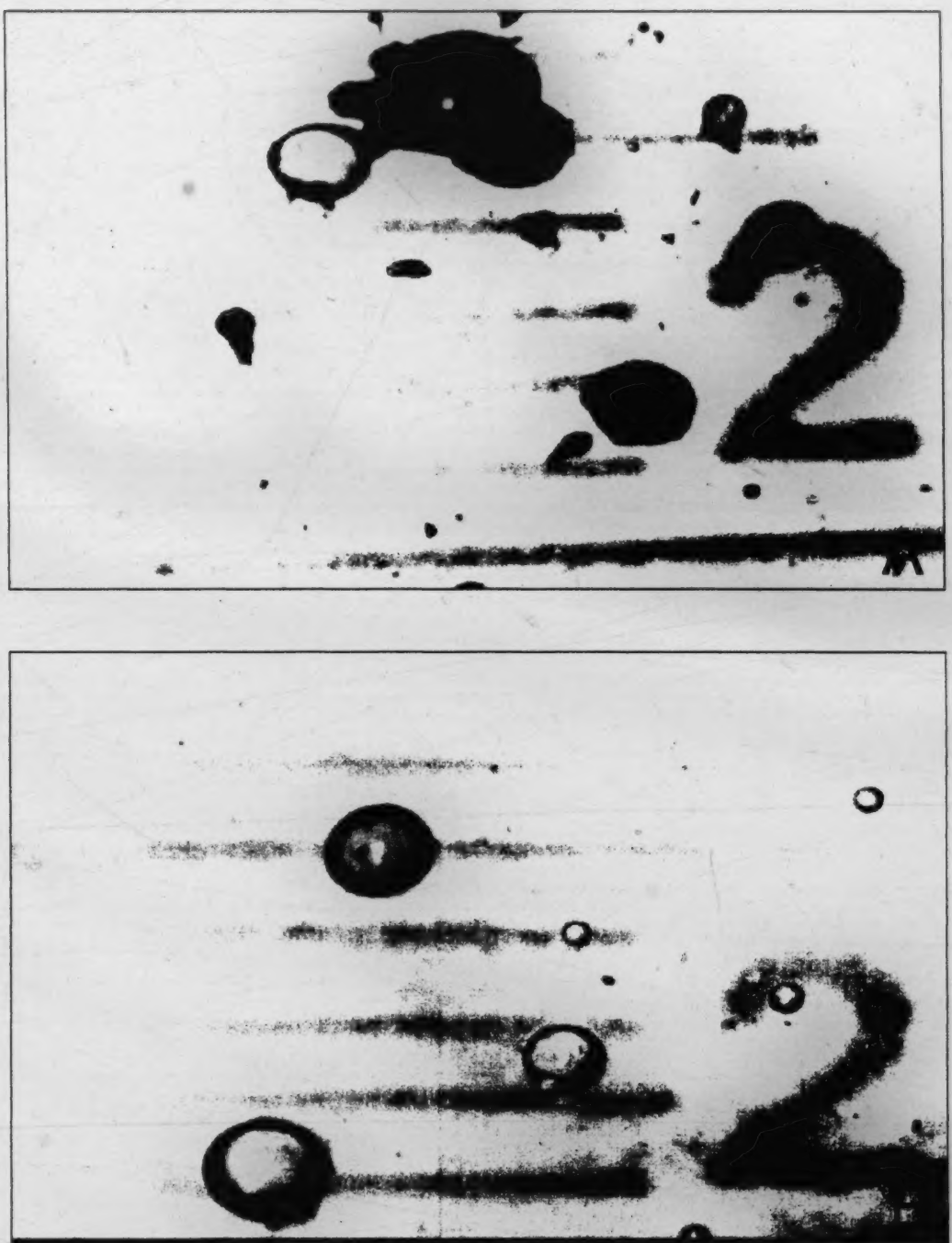

Figure 5-1 a) Bitumen laden bubbles at 50°C with 22% - 44 µm ore, good processing ore. b) Same conditions with 30% - 44 µm, poor processing ore.

5.2 Repeatability and Sensitivity

Using this newly constructed loop, tests were carried out on a typically "good" processing estuarine ore from Syncrude's North Mine, containing 12.3% bitumen and 22.1% solids in -44μm size fraction. Table B-3 contains the complete test conditions. The ore was processed with de-ionised water.

Experience has shown that the Batch Extraction Unit, when operated at lower temperatures can behave unpredictably. In contrast, Figure 5-2 shows that the kinetic bitumen recovery data is highly repeatable. The test conditions for the data sets in Figure 5-2 were a pipe velocity of 2 m/s at 50 °C. Air was added continuously at a rate of 150 sccm. This rate maintained an air to slurry volumetric ratio of 1:290 in the pipe, replenishing air that escaped through the open flotation container (Figure 4-1A, Figure 4-14, and Figure 4-19). Test 3 in this series was completed using a sample size of 1550 g as opposed to the standard 1000g sample. The results show that the system was insensitive to changes in sample size and thus slurry density over the range tested. Test 4 was performed with a flotation container approximately ¼ the size of the first three tests. The design change reduced the sample volume of the system. Again, the change in flotation container design did not impact the extraction characteristics of the ore. This finding indicates that the shape of the container for bitumen flotation shouldn't be a concern as long as it provides for a stable froth layer and opportunity for air bubbles to escape the slurry stream. The metal portions in the apparatus typically had about 5 to 10 grams of bitumen adhered to their surface, which

is not considered as froth product. That amount of bitumen accounted for 5 to 10 percent of the bitumen in the feed. After considering wall froth, recoveries over 90 percent by weight were achieved, which is comparable to commercial processes. Froth quality was also relatively consistent with average bitumen content of 46 ± 4 percent.

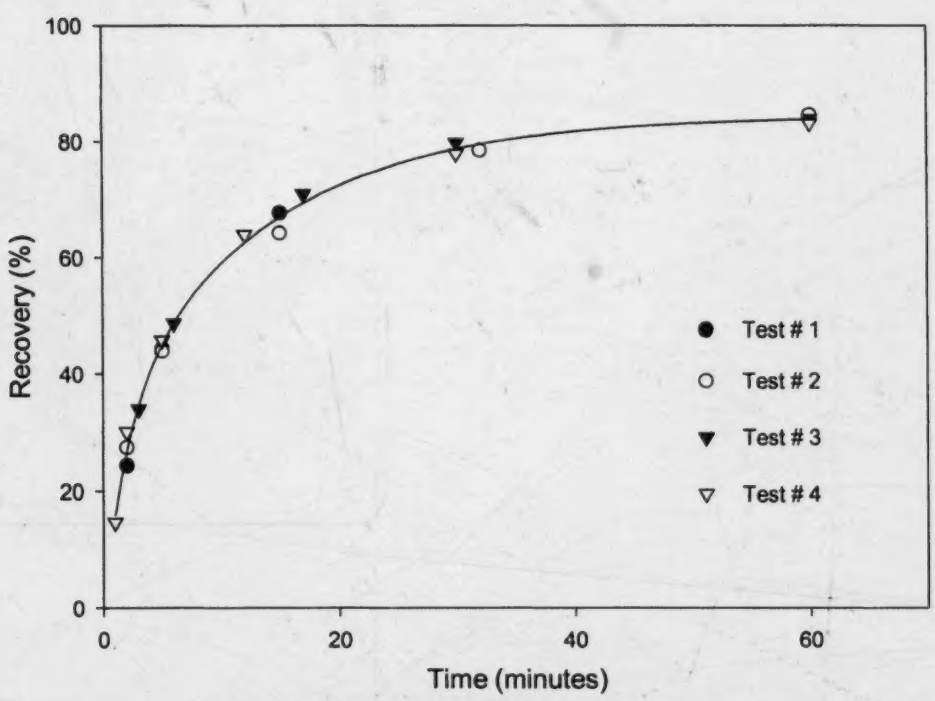

Figure 5-2 Bitumen recovery kinetics obtained in four sets of repeating tests.

5.3 Effect of Temperature on Bitumen Liberation

Experiments were carried out with a typical "good" processing ore with a grade of 14% bitumen and 15% solids in – 44μm size fractions and a typical "poor" processing ore with a grade of 9.5% bitumen and 30% solids in –

44μm size fraction. The tests were performed at a slurry velocity of 3m/s with varying air addition rates. Process water from Syncrude's base plant was used for tests in figures 5-3 through 5-9. The water had a pH of 8.0 and a conductivity of 4.07 mS. The ionic concentrations were 940 ppm HCO_3, 750 ppm chloride, 210 ppm sulphate and 17 ppm calcium. The slurried oil sands had a natural pH of 8.3. The slurry velocity was maintained at 3 m/s for all tests in figures 5-3 through 5-9. Details of the test configurations and data can be found in Appendix B.

At lower slurry temperatures, the viscosity of bitumen is extremely high, exhibiting a slow rate of bitumen liberation from sand grains. It is conceivable that highly viscous bitumen at lower temperatures slows down transfer of contained natural surfactants, such as naphthenic acids in bitumen to bitumen water interfaces, which is considered to increase the negative charge on both the bitumen and the sand grains, promoting the separation of the bitumen from the sand (Schramm, 1987).

From these two aspects, it is anticipated that lowering the temperature from 50°C to 35°C increases the time needed to liberate the bitumen from the solids as indeed observed in liberation results shown in Figure 5-3. For the low fines ores, for example, 50% of the bitumen was liberated after one minute at 50°C but it took five minutes at 35°C. A similar trend was seen for the high fines ores with 50% liberated after six minutes at 50°C and 21 minutes at 35°C, representing a 15 minute or 2.5 times increase in liberation

time. It is also evident that the liberation rate of the high fines ore is much slower and more sensitive to the reduction of temperature. After 60 minutes, bitumen was almost completely liberated from both ores. Analysis of the bitumen content in the tailings sand after 60 minutes confirmed the result of slurry image analysis that almost all of the bitumen had been separated from the solids. Comparison between Figures 5-3 and 5-4 shows a clear delay of bitumen flotation after liberation. As a result, the air attachment and flotation sub-process are identified as the rate limiting process in recovering bitumen from oil sands ores. The results also suggest that flotation is more effective for liberated bitumen than unliberated bitumen remaining on fines and sands.

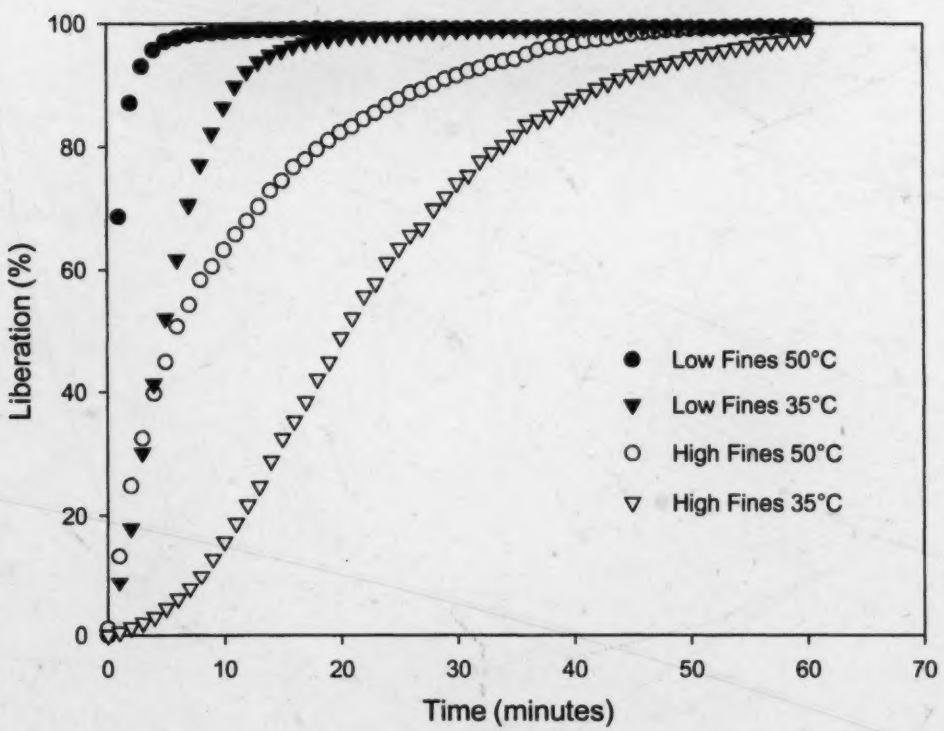

Figure 5-3 Effect of Temperature on Liberation

5.4 Effect of Temperature on Bitumen Recovery

Extensive research has shown that increasing temperature has a beneficial effect on bitumen recovery (Sury, 1990). It is well documented that the viscosity of bitumen varies dramatically with temperature. A viscosity drop by three orders of magnitude for bitumen has been reported as the temperature increases from 20°C to 80°C. At temperatures typically around 50°C and higher the bitumen has the tendency, after attachment, to spread and engulf the air bubble providing a very strong attachment. At lower temperatures, the bitumen attaches to air as a discrete particle with a much weaker binding through three-phase contact. It is well established that temperature affects the induction time, or time necessary for attachment between the bitumen and air to take place (Gu, 2002) as well as the rate of advancing three phase contact line, which is transferred to the strength of attachment. Temperature also influences the solubility characteristics of contained species in process water, influencing water chemistry, which may change the surface properties of the air bubbles and bitumen. Higher temperatures for example facilitate transfer/rearrangement of natural surfactants from bitumen to the interface. These surfactants aid bitumen-air attachment, improving the recovery of the bitumen. (Schramm, 1984)

Figure 5-4, obtained with an air to slurry ratio of 1:220 shows a similar impact of temperature on both the low fines ore and the high fines ore. In both cases, the overall bitumen recovery dropped by 25 to 30% when the temperature was reduced from 50°C to 35°C. Interestingly, the high fines

ore under the condition of 50°C showed nearly identical recovery characteristics as the low fines ore. This finding suggests that under favourable processing conditions some poor processing ores can be processed to achieve high recovery. It should be emphasised that it is possible to achieve high bitumen recovery from any ore so long as the correct "recipe" for processing that ore is found. In this example, a temperature of 50°C and an air addition at a rate of 1:200 air to slurry ratio were identified to be a suitable "recipe" for high recovery from this ore with our equipment. It is also interesting to note that high fines ore is more sensitive to lower temperature than lower fines ore.

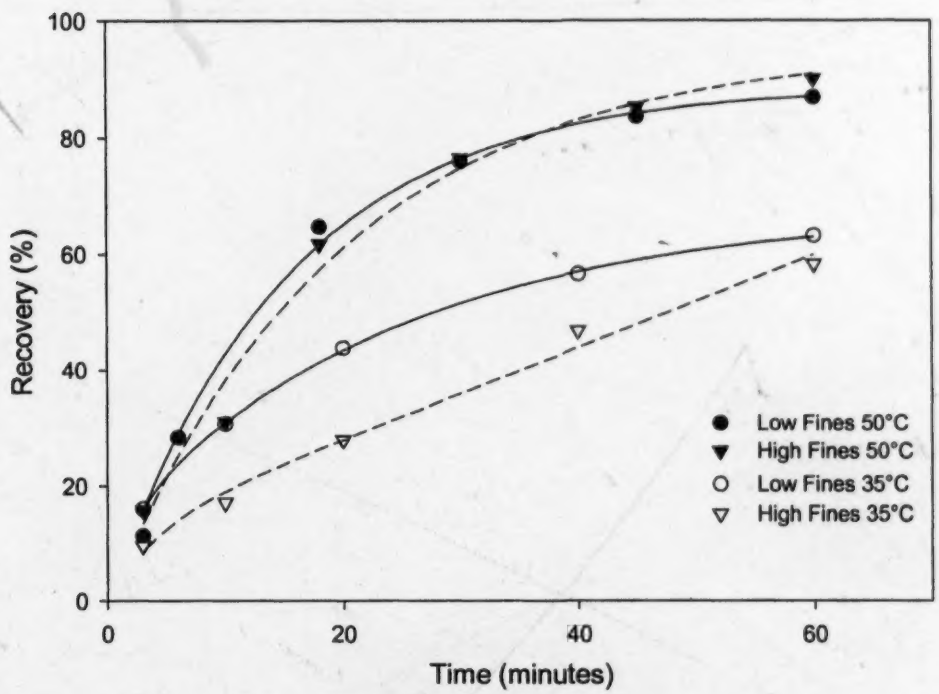

Figure 5-4 Effect of temperature on recovery

5.5 Effect of Air on Bitumen Recovery

The role of air in recovery of bitumen has been commented on in the literature. Sanford believed that the aeration step was the most difficult to achieve and poor bitumen aeration resulted in the most bitumen loss (Sanford, 1983). Clark also commented that air must be controlled within tight parameters to avoid excess mineral flotation to the froth (Clark, 1966). Studies on Syncrude's Aurora plant concluded that liberation of bitumen from sand grains was not problematic but actually getting the bitumen to float was the challenge to getting the process to achieve high recoveries (ManKowski, 1999)

As bitumen has the same density as water, in order to separate bitumen by flotation in a gravity separation vessel, bitumen has to be aerated. Although the role of air is much appreciated, little attention has been paid to the role of air availability in slurry conditioning. Sufficient air bubbles are needed to provide the necessary air bubble surface area to collect all the bitumen. Successful bitumen aeration provides an adequate density difference between the aerated bitumen and water for effective flotation in a gravity separation vessel. A series of experiments were carried out using the same high and low fines ores presented in the previous section. Air addition rates were changed to assess the impact the air:slurry ratio had on the recovery of bitumen to the froth. Since the attachment of bitumen to air bubbles is

probabilistic in nature, one would expect a higher air:slurry ratio to increase the chances of bitumen-air attachment and consequently to improve bitumen recovery. This would be the case only when the slurry conditions were conducive to air-bitumen attachment, which may not be always the case as will be discussed later.

The effect of air addition for the two types of oil sand ores on bitumen recovery is shown in Figure 5-5 for the low fines ore and in Figure 5-6 for the high fines ore. In the two cases, slurrying temperature of 35 °C was employed. For the case of low fines ore, an increase in the final bitumen recovery from 40 to 60% was achieved by the addition of air at an air to slurry volumetric ratio of 1:200 (200ml/min). For the case of high fines ore (Figure 5-6), an increase in the final bitumen recovery from 15 to 60%, was achieved, by the addition of the same air to slurry volumetric ratio of 1:200. It is evident that the improvement is much more dramatic for the higher fines ore than for the low fines ore. Figure 5-6 also shows that further increase in air addition showed in further increase in bitumen recovery, but to less extent, illustrated the reduced benefit with excessive aeration. For example, Figure 5-6 clearly shows that an air to slurry ratio of 1:87 has a minimal impact on further increasing bitumen recovery as compared to a ratio of 1:200.

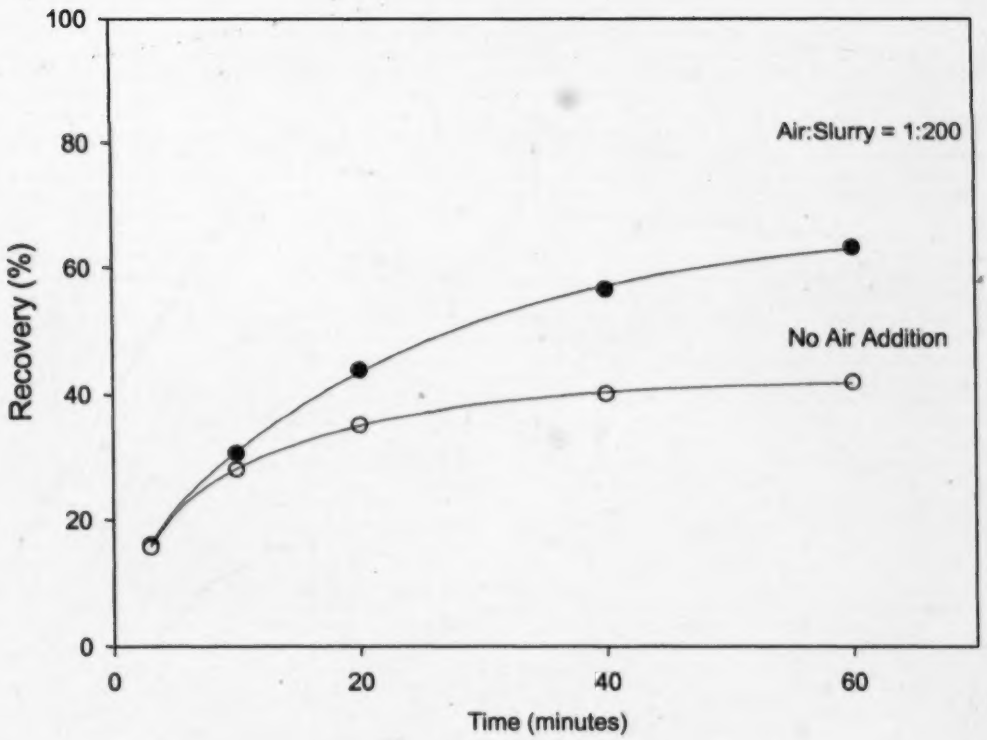

Figure 5-5 Effect of air on low fines ore at 35°C

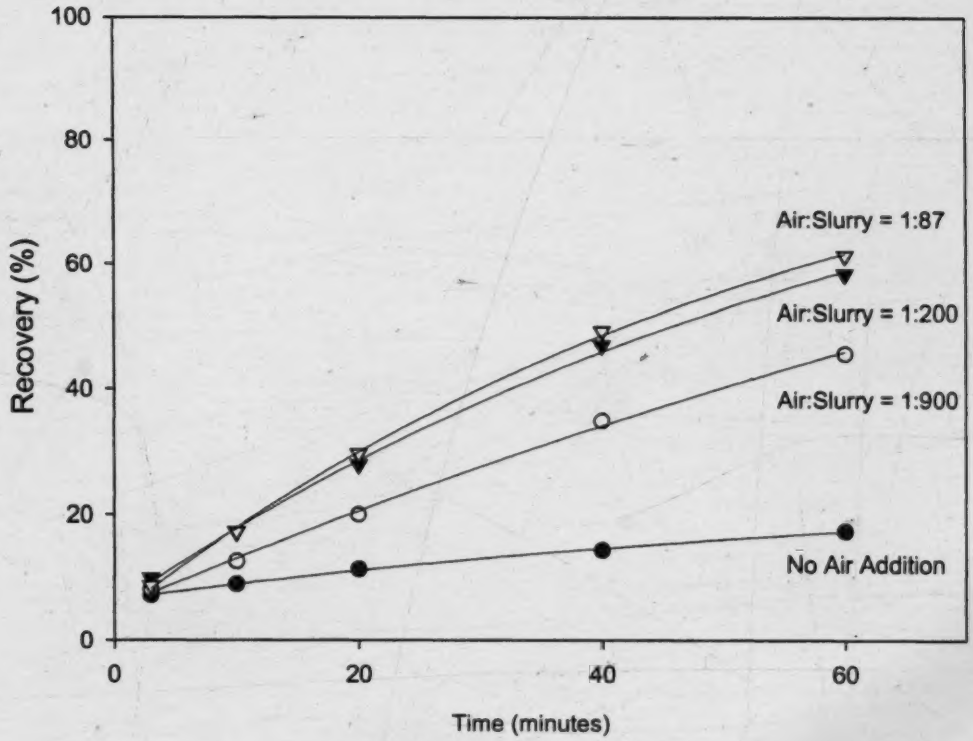

Figure 5-6 Effect of air on high fines ore at 35°C

Compared with bitumen recovery shown in Figure 5-6, Figure 5-7 shows that the liberation is generally quicker than flotation. It is clear that the difference in bitumen recovery can not be explained by an increased liberation rate. While the liberation does become faster, it is already much faster than the bitumen recovery rate. As shown in Figure 5-7, nearly 80 percent liberation is achieved in 20 minutes while bitumen recovery is only less than half of the liberated bitumen over the same period as shown in Figure 5-6. In all cases the liberation is significantly faster than the bitumen recovery.

From the results in Figure 5-7, it is evident that air has less influence on the liberation than bitumen flotation. Nevertheless, the results appear to suggest the benefits of air addition to increase the liberation rate, although to the limited extent. There are several possibilities that may explain this finding. First of all the increased air may in fact be attaching to bitumen and "pulling" if off the surface of the minerals. A second possibility is that more air in the system may increase the "white" area in the images, distorting the measurements of the image analysis program. Conversely the enhanced flotation rate due to the increased air rate may have been removing liberated bitumen droplets and perhaps even unliberated bitumen-mineral aggregates from the system, reducing the "black" area measured by the image analysis program compared to the base case with no air addition. In summary it is not believed that there is any real impact on true bitumen liberation through air addition, but that the results shown are most likely due to air influencing the liberation measurement.

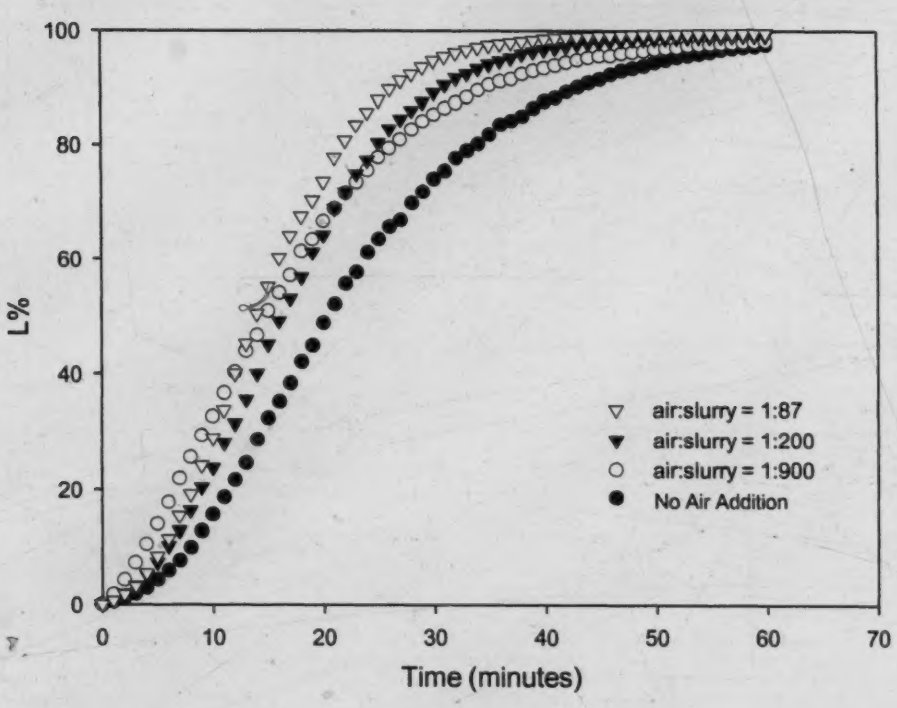

Figure 5-7 Effect of air on liberation of high fines ore at 35°C

To further test the role of air addition in bitumen recovery, a third ore known as "Transition" ore was tested (Figure 5-8). This Transition ore is from Syncrude Canada's Aurora mine and if found between the upper Esturian ore an lower marine ores in the Aurora mine pit. This ore is known to be difficult to process at low temperatures as experienced in commercial operations. When matched with unfavourable water chemistry, the contained clays may slime coat on bitumen, hindering air-bitumen attachment (Kasongo, 2000; Liu 2002). This ore has a grade of 8.8% bitumen by weight and 33% of contained solids are in -44μm size fraction. As shown in Figure 5-8 however, this ore exhibited excellent extraction

characteristics with appropriate air addition in our test loop. The continuous availability of fresh air bubbles in our loop test may account for the discrepancy between the LHES test results and commercial expectations. It is interesting to note that at an air addition rate of 50 ml/min (1:900 volume air to volume slurry flow) the accumulative amount of air added to the slurry is about three to one by volume. This air to bitumen volumetric ratio compares closely to a two to one ratio used in some commercial processes. The difference is that in our test, the air is added continuously as the slurry circulates and bitumen becomes liberated. This air addition pattern ensures a continuous supply of fresh bubbles available to attach to bitumen that have not been slime coated. Our results clearly show that air addition scheme is an important aspect to consider in engineering design of extraction processes. The implication is to have the minimum amount of air needed, available at the very beginning of and at staged intervals along a hydrotransport pipeline. The availability of fresh air bubbles at the time that bitumen is freshly liberated to ensure efficient aeration of bitumen may be a partial solution to the problem of clays and slime coating that interferes with bitumen aeration. The concept of staged aeration originally suggested by Bichard (1987) with regard to laboratory tests, applied throughout the length of a hydrotransport pipeline problems warrants further testing as a partial solution to slime coating.

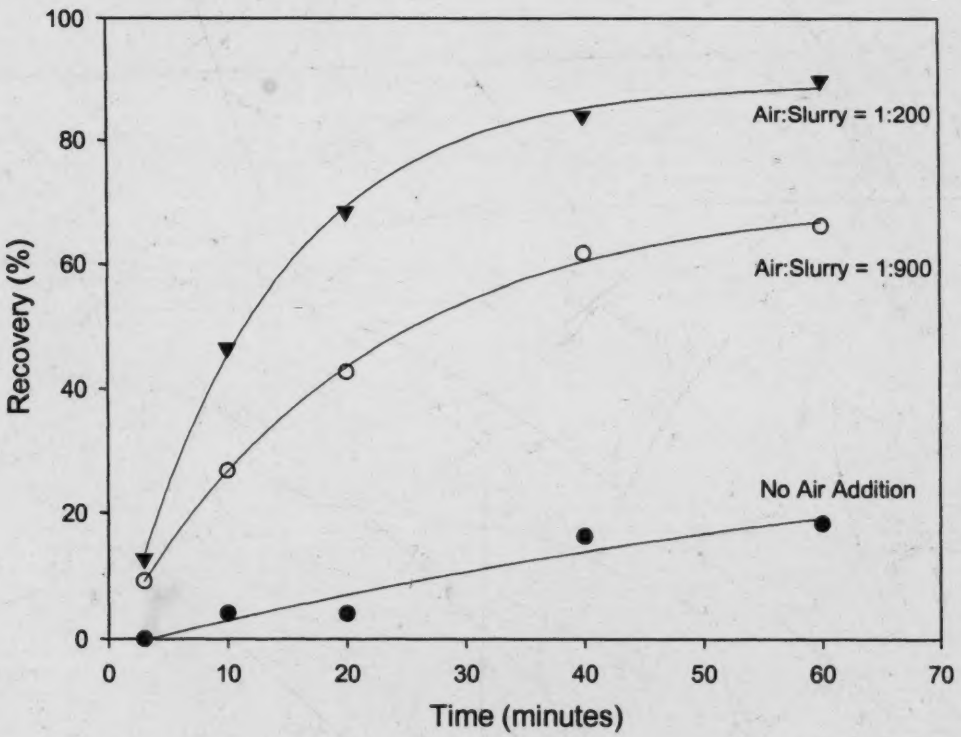

Figure 5-8 Effect of air addition on bitumen recovery from Transition ore at 35°C

We also tested with our loop a fourth type of ore, termed in the oil sands industry as oxidised ore that is also very difficult to process (Figure 5-9). This ore has a grade of 11% bitumen by mass and 13% of solids are in -44μm size fraction. The ore body has very shallow overburden and thus the ore has been exposed to oxygen for an extended period during which the bitumen has become severely oxidised. As a result, the bitumen surface becomes less hydrophobic and less amenable to air attachment. With this ore, air addition showed virtually no effect on bitumen recovery as seen in

Figure 5-9. This is one case where slurry conditions are not conducive to air-bitumen attachment. Enhancing favourable surface properties between air and oxidised bitumen is a research area currently being investigated by our research group.

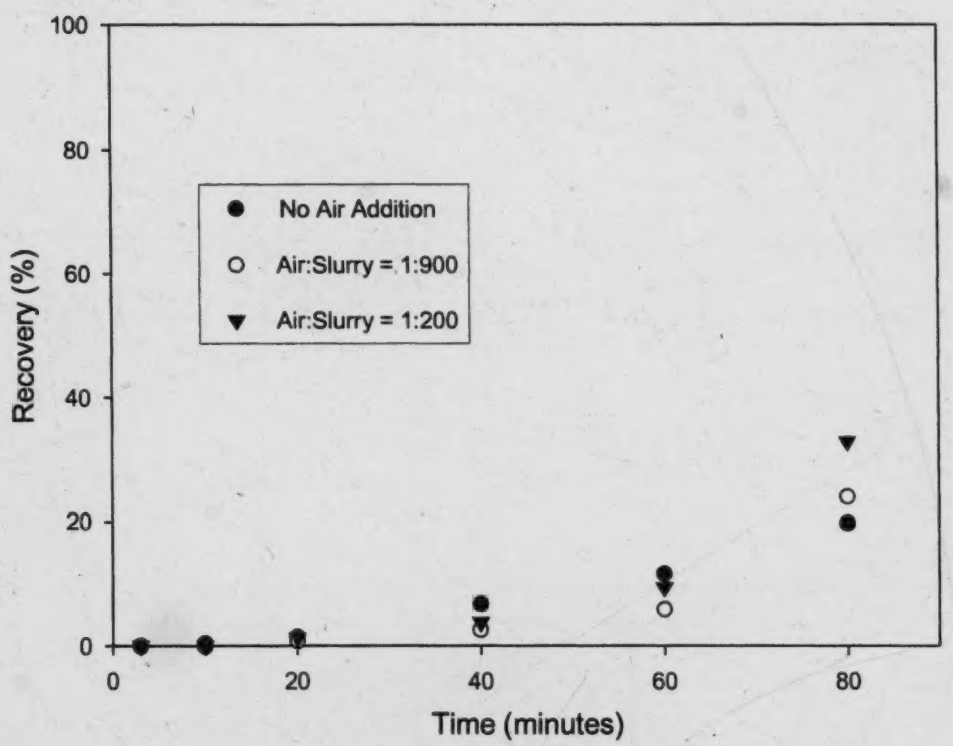

Figure 5-9 Effect of air on oxidised ore at 35°C

Solving the problem poor-processing ores such as those that are heavily oxidised or have high fines content requires understanding of the physical and chemical parameters of the system. Understanding the effect of fundamental parameters such as physical conditions, surface chemistry and

water chemistry is the true key to mitigate the difficulties encountered with these ores are present. Air addition may be one aspect of the physical parameters that can be managed to reduce the impact of difficult surface properties resulting from problematic ore composition and adverse water chemistry. In the future detailed analysis of water chemistry and surface properties of the slurry constituents would compliment the tests performed with the Laboratory Hydrotransport Extraction System.

Chapter 6 - Oily Bubble Flotation

Bitumen recovery from poor processing ores, often referred as oxidised ores and transitional ores has posed a great challenge to the oil sands industry. For such ores, bitumen recovery is in general low. The traditional addition of process aids, such as sodium hydroxide or sodium silicate is normally found ineffective to improve bitumen-air attachment and hence bitumen recovery. As well, results from the direct addition of light hydrocarbons, such as kerosene, directly to the oil sands slurrying liquid medium were found to be not encouraging.

In contrast to the oil sands industry, the mineral and coal process industries rely heavily on chemical additions to recover valuable minerals and or suppress unwanted minerals. Such chemicals are normally referred to as collectors or depressants, respectively. More over, liquid hydrocarbon addition to the air is utilised in the coal industry (Taggart, 1927; Drzymala and Wheelock, 1997; Shen and Wheelock, 2000, 2001) to recover coal fines. In this chapter kerosene addition to air bubbles to enhance bitumen-air attachment of poor processing ores and hence to improve bitumen recovery was studied. The tests were conducted using a specially constructed oil sands pipeline loop. Kerosene vapour was added into a flowing stream of air entering the slurry. The kerosene is anticipated to condense in air bubbles as they form in the slurry and migrate to the air bubble surface. We will refer to such air-kerosene bubbles as oily bubbles.

6.1 Oily bubble generation.

A variety of devices can be used to generate oily bubbles. The device used in this study is schematically shown in Figure 6-1. A controlled volume of kerosene is allowed to drip over a hot metal tube held at 200°C by an electric heating coil. Metered gas is allowed to flow in the heated tubing, carrying the vaporised/atomised kerosene. The gas-kerosene mixture is then injected into the pipeline loop via the port shown in Figure 4-1E and Figure A-4 using an 18-gauge needle. Visual observation confirmed that a thin layer of kerosene evenly coated the gas bubbles.

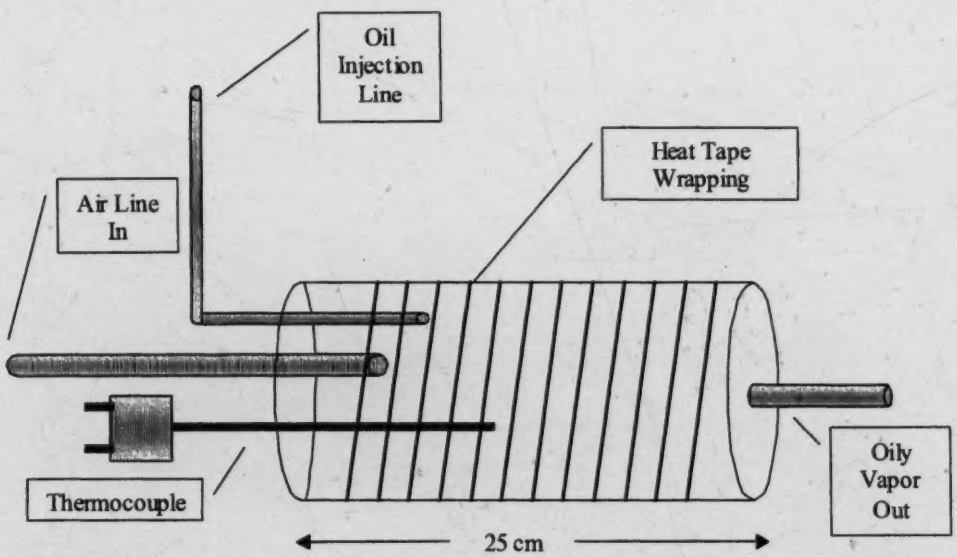

Figure 6-1 Schematic diagram of the apparatus used to generate air-kerosene bubbles or "oily bubbles"

It should be emphasised that kerosene is a highly flammable hydrocarbon and anytime air and hydrocarbons combine in high temperature environments there is a risk of fire and possible explosion. For this reason

tests such as these should be performed using an inert gas such as nitrogen in place of air. *Initial tests presented here were performed with air, but after reviewing the procedures from a risk and safety standpoint it is recommended that all future work be performed using an inert carrier.*

Alternate methods for mixing kerosene and air are also possible, although not tested here. Steam stripping of kerosene is one option, as is mechanical atomisation at room temperatures. Both of these methods have safety advantages over vaporisation at high temperatures.

6.2 Results

Two difficult-to-process Athabasca oil sand ores obtained from Syncrude Canada Ltd. and Suncor Energy Inc. were used in demonstration tests for bitumen extraction using oily bubbles. The oil sand charges were 1kg of ore together with 3kg of process water obtained from Syncrude Canada commercial oil sand plant. The ore from Syncrude was an esturarian ore with 9% bitumen content with 30 of solids in –44 micron mineral size fraction. The ore from Suncor was a heavily oxidised ore with a grade of 11.5% and 13% of solids in – 44 micron size fraction. This oxidised ore is known to be difficult to recover without high dosages of caustic addition. The slurry was circulated in the loop using a pump at a linear velocity of 3m/s. Air with or without kerosene was continuously injected into the slurry. The size of air bubbles generated as such was less than 1mm. Aerated bitumen was allowed to continuously float inside the open trough located in the pipeline

loop (Figure 4-1A). The floated bitumen in the chamber was collected at pre-determined time intervals. The amount of recovered bitumen at a given time was determined using Dean-Stark analytical method. Bitumen recovery kinetics were then established.

6.2.1 Control test

The Suncor oxidised ore was used in the control test. This ore contains approximately 11.5% bitumen by weight and is known in the industry as a heavily oxidised and it is hard-to-process ore. In this test, no kerosene was added to the injected air. Air was injected at a rate of 200ml/minute (air:slurry = 1:200). The test was carried out at 35°C. Bitumen recovery for the control test is shown in Figure 6-2 by open circles. It is evident that bitumen recovery was very poor, with less than 20% bitumen being recovered after 80 minutes of circulation inside the pipeline loop. A repeat control test (open triangles) shows poor flotation results akin to the first test.

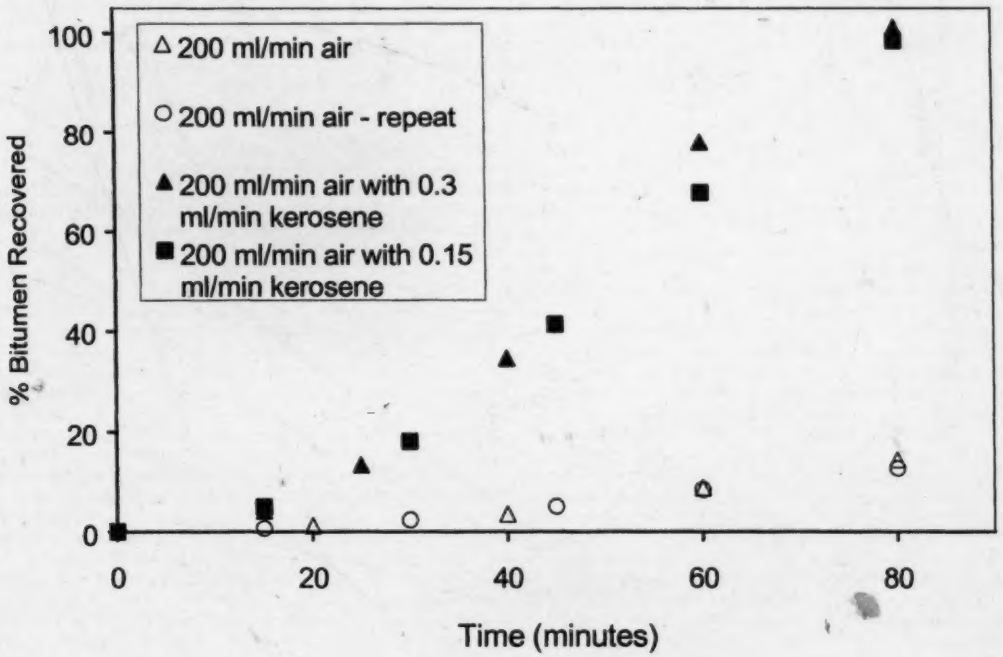

Figure 6-2 Variation of bitumen recovery with time: comparison of bitumen flotation using air and flotation using hydrocarbon (kerosene) aerosol of an oxidised, difficult to recover ore at 35°C.

6.2.2 Kerosene-Containing Air Bubble Test: Oily Bubbles

In this test, air was added at the same rate as in the control test, i.e., 200 ml per minute to the system. However, kerosene was added to the air prior to its injection into the slurry in the pipeline. The kerosene was added by vaporising 0.3 ml per minute kerosene at 200°C into the air stream. Using the device shown in Figure 6-1. Closed triangles in Figure 6-2 show a drastic increase in bitumen recovery kinetics with kerosene addition into the flowing air stream. After 80 minutes, nearly 100% bitumen recovery was obtained.

Bitumen recovery-time curve obtained for the oxidised ore with the kerosene-air addition becomes comparable to good processing ores. The results suggest that it is possible to increase bitumen recovery for hard-to-process ores, e.g., oxidised ores, to normal accepted levels that are comparable to "good" processing ores by using kerosene-coated air bubbles.

Using the same ore, a test was performed under the same conditions except for using a half amount of kerosene at a rate of 0.15 ml/min. The results shown as closed squares in Figure 6-2 confirmed as equally beneficial impact of oily bubbles on bitumen recovery.

A second difficult-to-process ore, was also tested at a lower processing temperature (23°C). This second ore contains a high fine content that is known to interfere with bitumen recovery process. The ore contains 30% of total solids in -44 μm size fraction and has a grade of approximately 9.5% by weight. The results are shown in Figure 6-3. After 60 minutes of circulation, use of oily air bubbles increased bitumen recovery from 20% to over 60% as compared to the case without kerosene addition as shown by the closed circles in Figure 6-3.

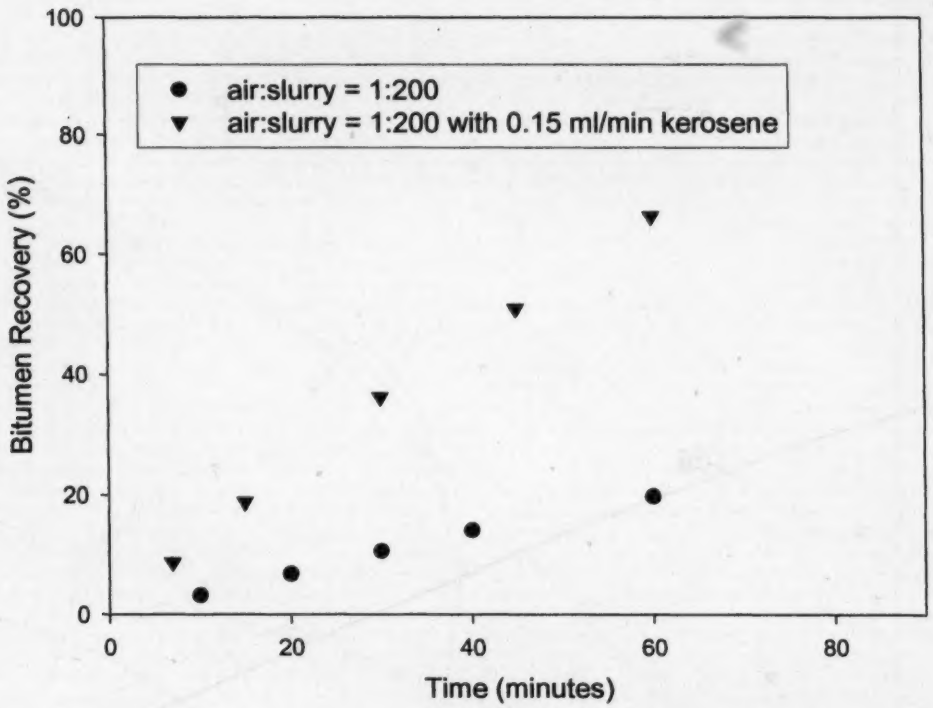

Figure 6-3 Variation of bitumen recovery with time: effect of hydrocarbon aerosol (kerosene) flotation of high fines, poorly processing ore at 23°C

Figure 6-4 shows that the liberation rate for the oxidised ore and the poor ore is much greater than the flotation rate without the use of oily bubbles. Kerosene addition showed a marginal increase in bitumen liberation. In contrast, a significant impact of kerosene addition on bitumen recovery was observed. In face, the rate of bitumen flotation with oily bubble is very close to that of the liberation rate, suggesting that the liberation rate is the limiting factor in these tests and that the oily bubbles are floating all the bitumen that becomes liberated.

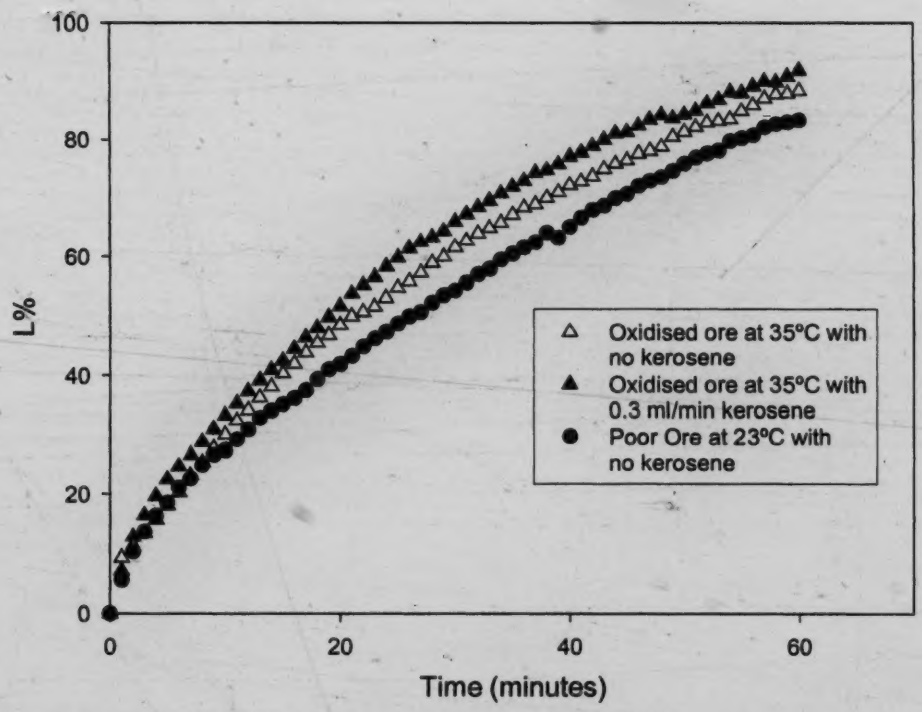

Figure 6-4 Variation of liberation of selected tests from figure 6-2 and 6-3 showing that kerosene addition to air does not significantly influence bitumen liberation rate

The mechanism for the substantial increase in bitumen recovery could be due to the alteration in the surface characteristics of the air bubbles. The vaporised kerosene into the flowing air stream would condense on the air bubble surface when the air is contacted with the slurry. The micron-thick kerosene at the air-slurry interface acts as a good collector for the liberated bitumen. Attachment of the air bubbles to the bitumen is enhanced due to the presence of the kerosene film.

Although understanding the mechanistic role of kerosene in enhancing bitumen flotation requires further study, using kerosene to assist air/bitumen attachment is conceptually a valid approach. In fine coal flotation, kerosene is known to spread readily on coal surfaces upon making contact with coal over hydrophobic surface patches/domains. The subsequent spreading of kerosene on coal masks exposed hydrophilic domains, rendering entire coal surface hydrophobic and resulting in an overall stronger air-coal attachment for flotation. Recently, Xu et al. 2002 and Liu et al., 2002 measured contact angle of kerosene containing air bubbles on sulphide minerals in water. Compared with the maximum attainable contact angle values (<100°) of air bubbles on mineral surfaces, a much higher contact angle value (160°) was observed for air bubbles covered with a thin layer of kerosene containing potassium dodecyl xanthate, suggesting a stronger air-mineral attachment. Bitumen extraction is an extremely complex physicochemical system. On one hand, various types of surfactant are known to release into the process water. These surface active molecules accumulate at air/water interface more effectively than at kerosene/water interface due to a higher potency of interfacial energy (72 mJ/m^2) for air/water interface than that (50 $mJ/m^{2)}$ for kerosene/water interface. As a result, the surfactant-covered air bubble surface is more rigid compared with kerosene surface in water. Bitumen, on the other hand, is known to be highly soluble in kerosene and hence a reduced viscosity at the contact region can be achieved. Kerosene film on an air bubble therefore assists the mobility of surface contact area and thereby

enhances the spreading kinetics within the contact area and hence flotation kinetics. A fundamental understanding of the improved bitumen recovery by using oily bubbles would lead us not only to develop this particular new process for effective bitumen recovery from poor processing oil sands ores, but also would suggest that other type of low viscosity water-immiscible liquids can be used in this process, so long as the liquid itself has some degree of solubility for bitumen.

6.3 Discussion

Kerosene at a ratio of 0.00075 liquid additive to air was found to be sufficient for the tests conducted in this work. This ratio is not optimised but is in the general range required to be effective. In oil sands the air addition rate can be between 5% and 20% (sometimes higher) by volume in a hydrotransport pipeline. This would equate to about 100 – 400 ppm of additive per tonne of oil sand in a hydrotransport pipeline. This rate is similar to rates used in commercial applications when additives were injected directly into the slurry. The increased efficiency of the additives when added directly to the air bubbles and thus delivered directly to the interface where the additives are expected to enhance air-bitumen attachment should be significant. One requirement for the effective application of oily bubbles is that there is exposed oily air bubble present to attach to bitumen droplets. If the oily bubbles attach to bitumen and thus become bitumen engulfed air bubbles then the effectiveness of the oily bubbles becomes nullified. The may mean that oily bubbles would be less effective in environments with significant

bitumen present, such as hydrotransport lines. It could be hypothesised that in a hydrotransport environment the majority of bitumen aeration occurs by which already aerated bitumen droplets attach to other bitumen droplets through a bitumen-bitumen interface. In other applications such as flotation cells and secondary recovery applications the proportion of air bubble surface area to bitumen is much greater. In these applications, where the scarcity of bitumen already decreases the probabilistic chance of air-bitumen contact and attachment the enhanced efficiency of an oily bubble may be most effective. The challenge in these applications is that the volume of air used can sometimes be significant and thus the amount of additive required can be challenging.

Some of the potential application points for oily bubble technology would be the Tails Oil Recovery (TOR) vessels at Syncrudes Base Plant. Oily bubble injection into the feed stream of these vessels may be lucrative. Another potential application is for sparging of oily bubbles into the cone of primary separation vessels or cells, particularly on vessels such as those at Syncrude's Aurora plant where there is no additional recovery processes for tailings. Column flotation cells may also present a potential application of oily bubble technology. Column flotation is not currently applied in oil sands at this time, but research and piloting is underway and column cells could potentially be applied in the near future.

Chapter 7 - Conclusions

A novel apparatus was developed for testing oil sand processability under hydrotransport-based conditions of variable temperatures. This equipment has been shown to be repeatable and sensitive to processing conditions, while being insensitive to density and flotation vessel configurations. For the first time the liberation and recovery kinetics have been analysed, independently, yet concurrently, of one another. The following conclusions can be drawn based on the experimental results:

Bitumen liberation rate, in general, is faster than the flotation rate, making air attachment and subsequent flotation sub-processes as the rate-limiting step.

Oil sand processing at lower temperatures has a negative effect on bitumen liberation. The effect appears to be consistent for both high and low fines ores. This result is likely due to the effect of temperature on bitumen viscosity and the transport of natural surfactants that influence the surface properties of the bitumen and sand grains.

Lower operating temperatures result in a lower bitumen recovery. This is likely the result of poor air-bitumen attachment. Lower temperatures have the effect of increasing the induction time needed to attach air to bitumen and hence decrease the probability of attachment upon air bubble-bitumen collision. Additionally, a lower processing temperature leads to an increased bitumen viscosity. In turn, a high bitumen viscosity drastically slows the

expansion of the three-phase contact line and the resulting attachment strength between the bitumen and air.

Air addition improves the recovery of both low fines and high fines ores, provided that the bitumen is not highly oxidised. High fines ores experience a greater degree of recovery improvement due to air addition than low fines ores. The continuous supply of fresh bubbles shows potential for partially overcoming clay slime coating problems associated with some ores. This finding supports a staged aeration strategy for commercial operations.

Only a relatively small air to slurry ratio is needed for maximum impact and further air addition has diminishing returns in terms of improved recovery.

Addition of kerosene to air bubbles for bitumen recovery in a laboratory pipeline was found to be highly beneficial for improving bitumen recovery from poor processing ores.

Chapter 8 - Recommendations and Future Work

8.1 Recommendations

8.1.1 Experimental apparatus

In this study the temperature control was managed manually. For better accuracy a larger heat exchanger section and a higher capacity heater/chiller with a fast response time and under computer process control would improve temperature control.

A taller loading/flotation vessel with a narrower neck would allow faster pipeline velocities while maintaining a quiescent froth layer. Baffles may also improve froth layer stability.

Addition of a gate or blind to isolate the slurry flow from the flotation vessel would be useful for experiments whereby the slurry is conditioned fully as in a commercial hydrotransport line before being permitted to separate.

8.1.2 Study air bubble size

Through the use of the bubble visualisation tube the study of air bubble size could be enlightening in determining flotation efficiency of bitumen. As well parameters such as air addition rates and methods could also be studied.

8.1.3 Mechanical energy effects

By varying the flow rate through the loop the impact of mechanical energy on bitumen flotation could be achieved. Examination of bitumen liberation

versus flotation with attention being paid to the rate limiting step may prove useful in determining required conditions for efficient bitumen extraction and provide some guidance to the required mechanical energy needed to fully liberate bitumen from different oil sands.

8.1.4 Viscosity impact on flotation

The doppler flow meter installed on the LHES depends on a user (or in most cases manufacturer) specified fluid viscosity to calculate the velocity based on the doppler reflections. Generally this is set to the viscosity of water. In the case of oil sand slurries this complicates the use of doppler meters for velocity measurements since the viscosity of a oil sand slurry in never constant. It changes with varying water chemistry, temperature and ore type. As an oil sand is slurried and conditioned in a hydrotransport line the water chemistry changes as the ore and water become intimately mixed. In the case of the LHES as bitumen is removed through flotation the composition of the slurry changes and thus the viscosity. This can be seen in the measurements taken by the doppler flow meter. In the beginning of a test when the oil sand is mixed with the water the flow meter tends to underestimate the velocity in the system. As the slurry is conditioned an as the bitumen is separated the flow meter measurement increases until the measurement is closer to that of just water. Using a positive displacement pump that is in good condition and thus knowing the true velocity, the equations used in the doppler flow meter could be used to back calculate the

viscosity of the system. This would provide a measurement of in-situ viscosity that previously was unavailable.

References

Alberta Energy and Utilities Board, "Crude Bitumen Reserves Atlas", Alberta Energy and Utilities Board Statistical Series, **96**, No. 38, 169p. (1996)

Alexander, K.L., and Li, D., "Effects of bitumen films over air bubble surfaces on bitumen drop-air bubble attachment". Colloids and Surfaces A: Physicochem. And Engineer. Aspects, **106**, 191-202, (1996).

Bichard, J.A., "Oil Sands Composition and Behavior Research", AOSTRA Technical Publication Series #4, Alberta Oil Sands Technology and Research Authority, Edmonton, AB (1987).

Bulmer, J.T., and Starr, J., "Syncrude Analytical Methods for Oil Sand and Bitumen Processing", The Alberta Oil Sands Technology and Research Authority, 173p. (1979)

Clark, K.A., "Athabasca oil sands – Fundamentals affecting development", Can. Pet., **7**, 18-21 (1966).

Clark, K.A., AND Pasternack, D.S., Industrial and Engineering Chemistry, **24**, 1410-1416 (1932)

Clark, K.A., and Pasternack, D.S., Research Council of Alberta, Report No. 53, University of Alberta, Edmonton, (March, 1949)

Clark, K.A., CIM Transactions, XLVII, 257-274 (1944).

Czarnecki, J., Liu, J.K., Smith, R.G., and Tyerman, W.J.R., "Extraction of bitumen from Athabasca oil sands: hot water process theory, addressing

temperature effects, in Can. Chem. Eng. Conf, Vancouver, BC, **1**, (1991).

Dai, Q., Chung, K.H., "Hot water extration process mechanism using model oil sands", Fuel, **75**, 220-226, (1996).

Drelich, J., Lelinski, D., and Miller, J.D., "Microscopic observations of bitumen spreading at gas bubble surfaces". Processing of hydrophobic minerals and fine coals: Proceedings of the 1st UBC-McGill bi-annual international symposium on fundamentals of mineral processing, Laskowski, J.S., and Poling, G.W., ed(s)., Can. Inst. Mining, Metallurgy and Petroleum, Vancouver, BC, 291-304, (1995).

Drelich, J., Lelinski, D., Hupka, J., and Miller, J.D., "Comparison of water-based physical separation processes for U.S. tar sands". Fuel, **74**, 1150-1155, (1995).

Drzymala, J. and Wheelock, T.D., "Air-Promoted Oil Agglomeration of Moderately Hydrophobic Coal. 2. Effect of Air Dosage in a Model Mixing System"., Coal Preparation, **18**, 37-52, (1997).

FTFC (Fine Tailings Fundamentals Consortium), Advances in Oil Sands Tailings Research, Alberta Department of Energy, Oil Sands and Research Division, Edmonton (1995).

Gillespie, T., and Rideal, E.K., "The coalescence of drops at an oil-water interface". Trans. Faraday Soc., **52**, 173-183, (1956).

Grant, G.B. Parameters and mechanisms in the mechanical upgrading of Athabasca oil sands by a cold water process. M.Sc. thesis, University of Toronto, 136 pp. (1977)

Guoxing Gu, Zhenghe Xu, Kumar Nandakumar and Jacob Masliyah, "Effects of physical environment on induction time of air-bitumen attachment". International Journal of Mineral Processing, 69, 235-250, 2003

Hepler, L.G. and C. Hsi, "AOSTRA Technical Handbook on Oil Sands, Bitumens and Heavy Oils", AOSTRA Technical Publication Series #6, Alberta Oils Sands Technology and Research Authority, Edmonton, AB (1989).

Hepler, L.G. and R.G. Smith, "The Alberta Oil Sands: Industrial Procedures for Extraction and Some Recent Fundamental Research", AOSTRA Technical Publication Series #14, Alberta Oil Sands Technology and Research Authority, Edmonton, AB (1994)

Innes, E.D. and Fear, J.V.D Canada's first commercial tar sands development, in "7th World Petroleum Congress", Elsevier, Mexico, 633-650, (1967).

Kasongo, T., Zhou, Z., Xu, Z., Masliyah, J., "Effect of Clays and Calcium Ions on Bitumen Extraction from Athabasca Oil Sands Using Flotation", Can J. Chem. Eng. **78**, 674-681 (2000).

Kasperski, K.L., "A Review of Properties and Treatment of Oil sans Tailings", AOSTRA J. Research **8**, 11-15 (1992).

Lam, W.W., Payette, C., MacComachie, C.A., Munoz, V.A., and Mikula, R.J., "Flotation of oil sands. Part I: process parameters and characterisitics", CRL 92-12 (CF), 1-67, Natural Resources Canada, Devon, AB, (1992).

Liu, J., Xu, Z., and Masliyah, J., "Studies of bitumen-silica interaction in aqueous solutions by AFM", Langmuir, 19, 3911-3920 (2003)

Luthra, M., "A Visualization Technique for Estimating Bitumen Recovery from Oil Sands", M. Sc. Thesis, University of Alberta, Canada (2001).

Masliyah. J.H., "Extraction and Upgrading of Oilsands Bitumen – Intensive Short Course", University of Alberta, (2002).

McKay, A. "Fundamentals of mechanical upgrading: mechanisms of sand and bitumen extraction form oil sands." M.Sc. thesis, University of Toronto, 117 pp. (1978)

McKay, A.M. and Phillips, C.R. and Sarkar, S. Mechanical upgrading of Athabasca oil sands in a rotating contactor. Ind. Eng. Chem Process. Des. Dev. **19**, 185-189 (1980)

Okada, K., Akai, Y., Kogure, M., and Yoshioka, N., "Effect of surface charges of bubbles and fine particles on air flotation process". Can. J. Chem. Eng., **68**, 393-399, (1990).

Sanford, E.C., "Processibility of Athabasca Oil Sand: Interrelationship between Oil Sand Fine Solids, Process Aids, Mechanical Energy and Oil Age after Mining", Can. J. Chem. Eng. **61**, 554-567 (1983)

Sanford, E.C., Seyer, F.A., "Processibility of Athabasca tar sand using a batch extraction unit: the role of NaOH". CIM Bulletin, **72**, 164 –169 (1979).

Schramm, L.L., R.G. Smith and J.A. Stone, "On the Processibility of Mixtures of Oil Sands", AOSTRA J. Res. **1**, 147-161 (1985)

Schramm, L.L., R.G. Smith and J.A. Stone, "The Influence of Natural Surfactants Concentration on the Hot Water Process for Recovering Bitumen from Athabasca Oil Sands", AOSTRA J. Res. **1**, 5-13 (1984)

Schramm, L.L., R.G. Smith, "Two Classes of Anionic Surfactants and their Significance in Hot Water Processing of Oil Sands", Can. J. Chem. Eng. **65**, 799-811 (1987)

Sepulveda, J.R., and Miller, J.D. "Separation of bitumen from Utah tar sands by a hot water digestion-flotation technique", Mining Engineering, **30**, 1311-1320, (1978).

Shen, M. and Wheelock, T.D., "Coal Agglomeration with Microbubbles", Coal Preparation", **21**, 277-298 (2000).

Shen, M. and Wheelock, T.D., "Development and Scale-up of a Gas-Promoted Oil Agglomeration Process for Coal Beneficiation", Minerals and Metallurgical Processing, **18** (2), 87-94 (2001).

Sury, K.N. "Low Temperature Bitumen Recovery Process", US Patent # 4,946,597, (1990)

Sury, K.N., Paul, R., Dereniwski, T.M., and Shulz, D.G., Next generation oilsands technology: the new OSLO processes, in "Oil sands

conference", Alberta Chamber of Resources, AOSTRA, EMR Canada, Edmonton, 1-36, (1993).

Taggart, A .F., "Handbook of Ore Dressing", John Wiley and Sons, New York, P. 945 (1927).

Takamura, K., and Chow, R.S., "The electrical properties of the bitumen/water interface. Part II: Appplication of the ionizable surface-group model". Colloids and Surfaces, **15**, 35-48, (1985).

Takamura, K., and Wallace, D., "The pysical chemistry of the hot water process". J. Can.Pet.Tech., **27**, 998-1068, (1988).

Tyerman, W.J.R., "Rold of dynamic properties in bitumen extraction from Athabasca oil sands", Processing of hydrophobic minerals and fine coals: Proceedings of the 1st UBC-McGill bi-annual international symposium on fundamentals of mineral processing, Laskowski, J.S., and Poling, G.W., ed(s)., Can. Inst. Mining, Metallurgy and Petroleum, Vancouver, BC, 329-338, (1995).

Wallace, D., Henry, D., and Takamura, K., "A physical chemical explanation for deterioration in the hot water processability of Athabasca oil sand due to aging". Fuel Sci. and Tech. Int., **7**, 699-725, (1989).

Wallwork, V.W., Z. Xu and J. Masliyah, "Bitumen Recovery with Oily Air Bubbles", Can. J. Chem. Eng., in press, (2003).

Wu, X., Czarnecki, J., Hamza, N., and Masliyah, J., "Interaction forces between bitumen droplets in water". Langmuir, **15**, 5244-5250, (1999).

Yoon, R.H., and Luttrell, G.H., "The effect of bubble size on fine particle flotation". Mineral Processing and Extractive Metallurgy Rev., **5**, 101-122, (1989).

Zhou, A.A., Z. Xu, J. Masliyah, T. Kasongo, D. Christendat, K. Hyland, T. Kizior and d. Cox, "Application of On-Line Visualization to Flotation Systems", Proceedings, 32nd Canadian annual Meeting of Mineral Processors, Ottawa, ON (2000).

Zhou, Z.A., Hussein, H., Xu, Z., Czarnecki, J., and Masliyah, J.H., "Interaction of ionic species and fine solids with a low energy hydrophobic surface from contact angle measurement". J. Colloid Int. Sci., **204**, 342-349, (1998)

APPENDIX A: PICTURES OF EXPERIMENTAL EQUIPMENT (LHES)

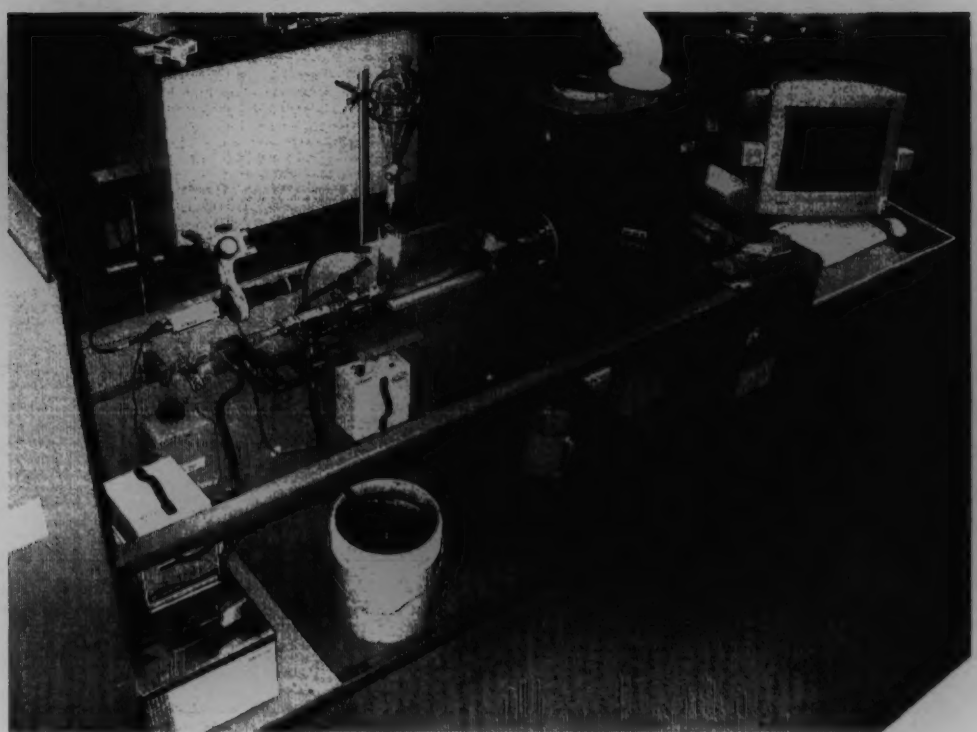

Figure A-1 Photograph of Laboratory Hydrotransport Extraction System

Figure A-2 Picture of LHES

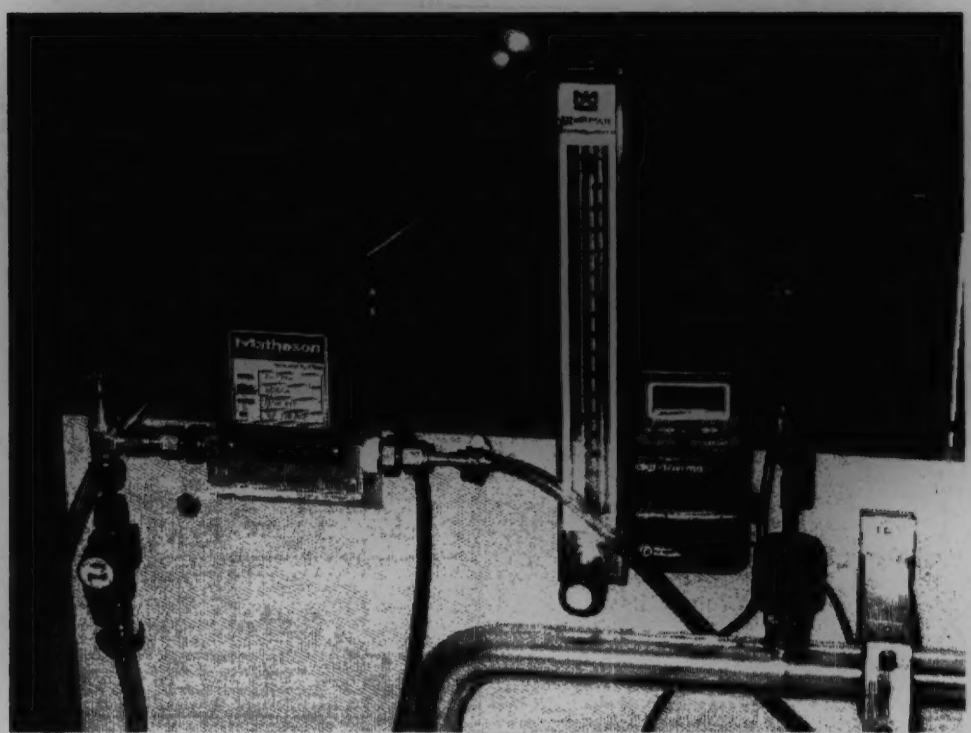

Figure A-3 Close up of gas metering instrumentation and temperature probe

Figure A-4 Close up of lower pump connections, lower drain valve, pressure relief valve and air injection point.

Figure A-5 Photograph of oily bubble generator

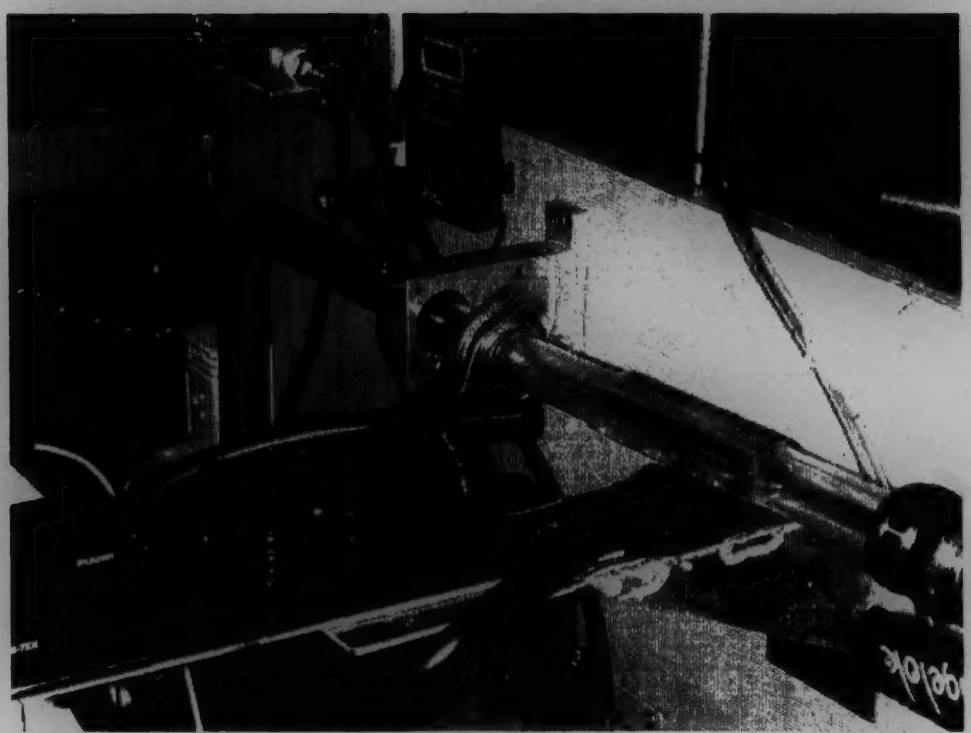

Figure A-6 Picture of slurry visualisation camera and squared section of glass tubing.

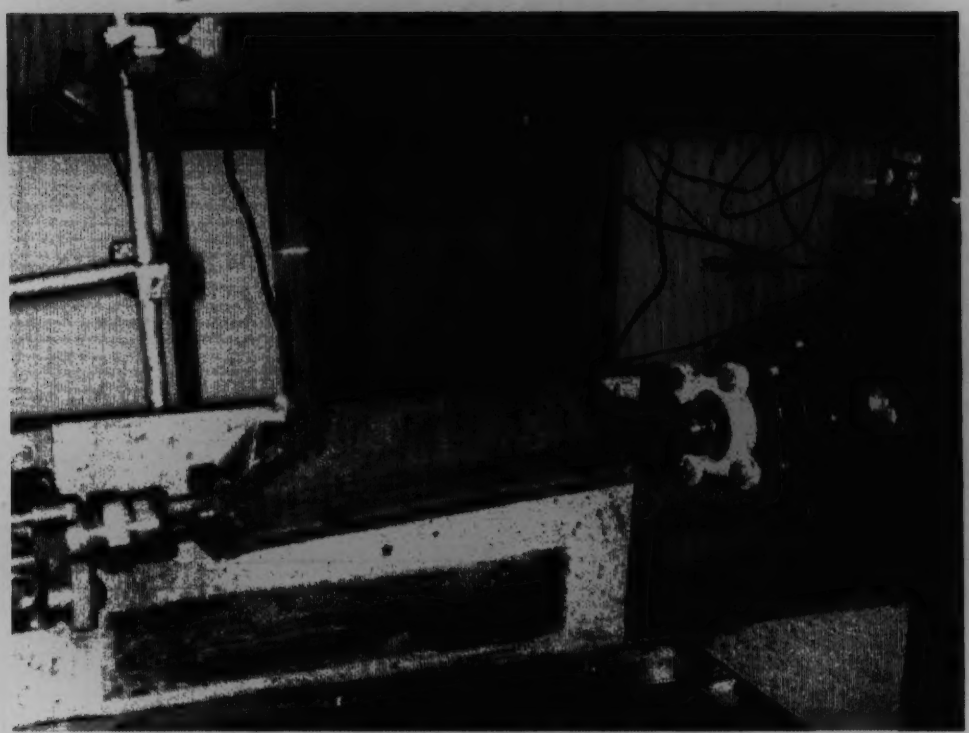

Figure A-7 Close up of loading/flotation vessel

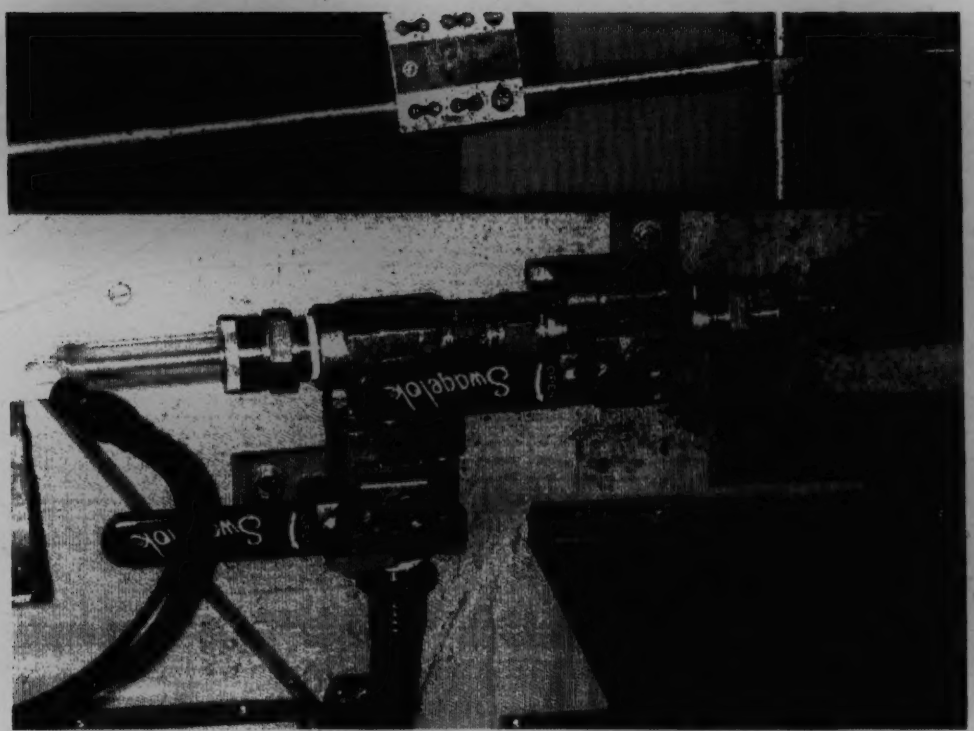

Figure A-8 Close up of valve assembly

Figure A-9 Close up of doppler flow meter transducers

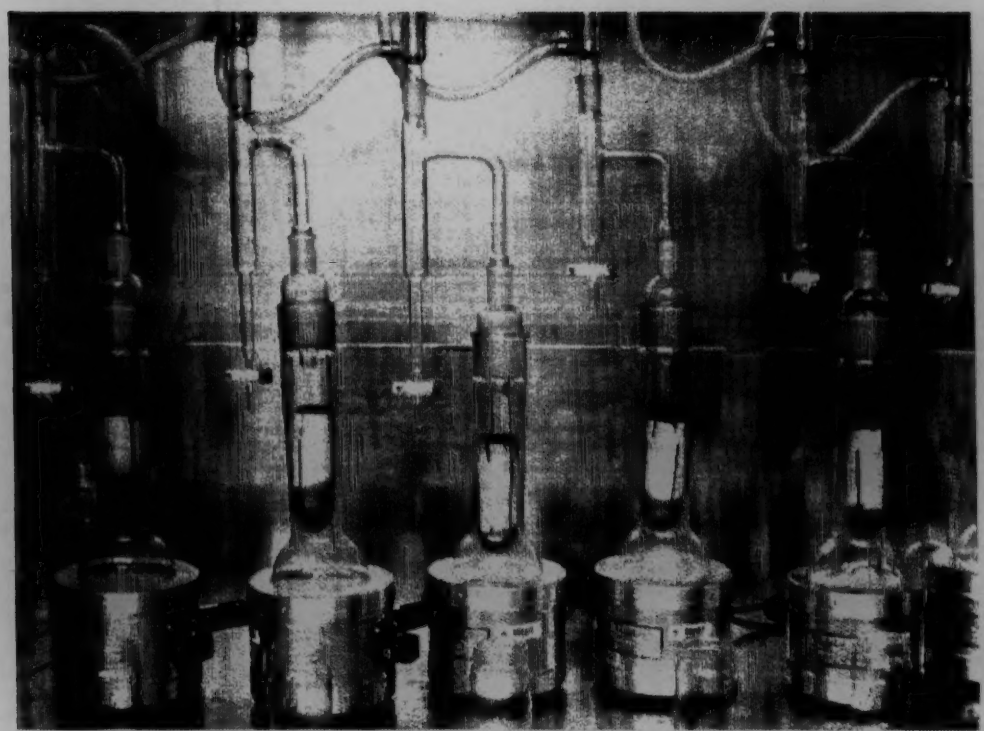

Figure A-10 Photo of Dean Stark separators for oil/water/solids analysis

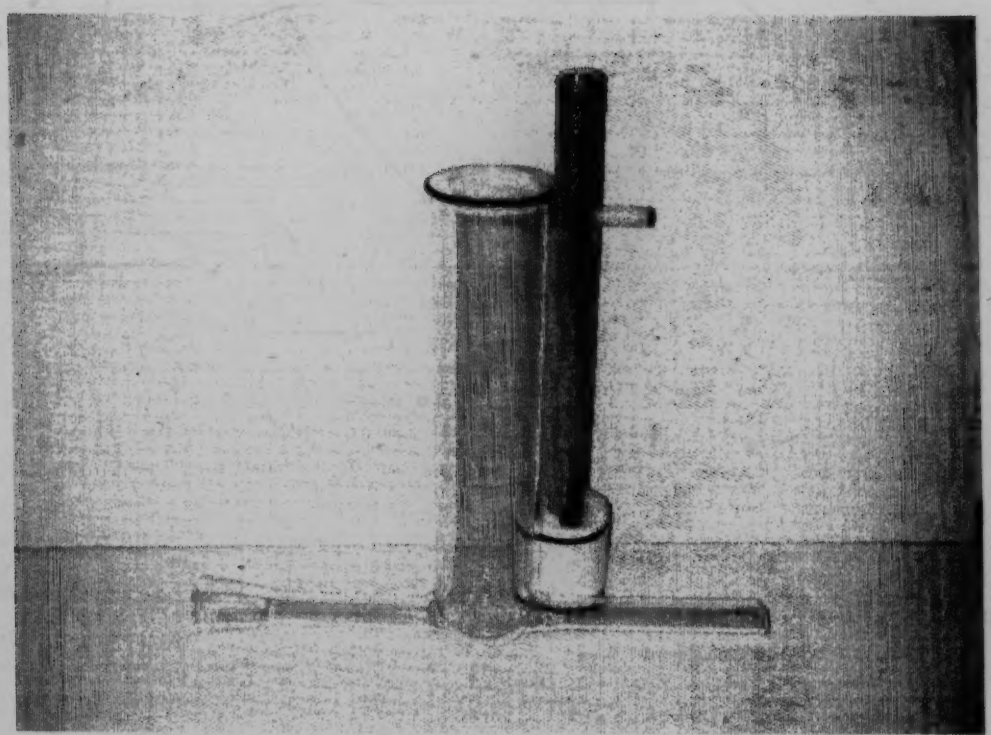

Figure A-11 Alternate loading vessel for closed loop tests (full batch mode procedure)

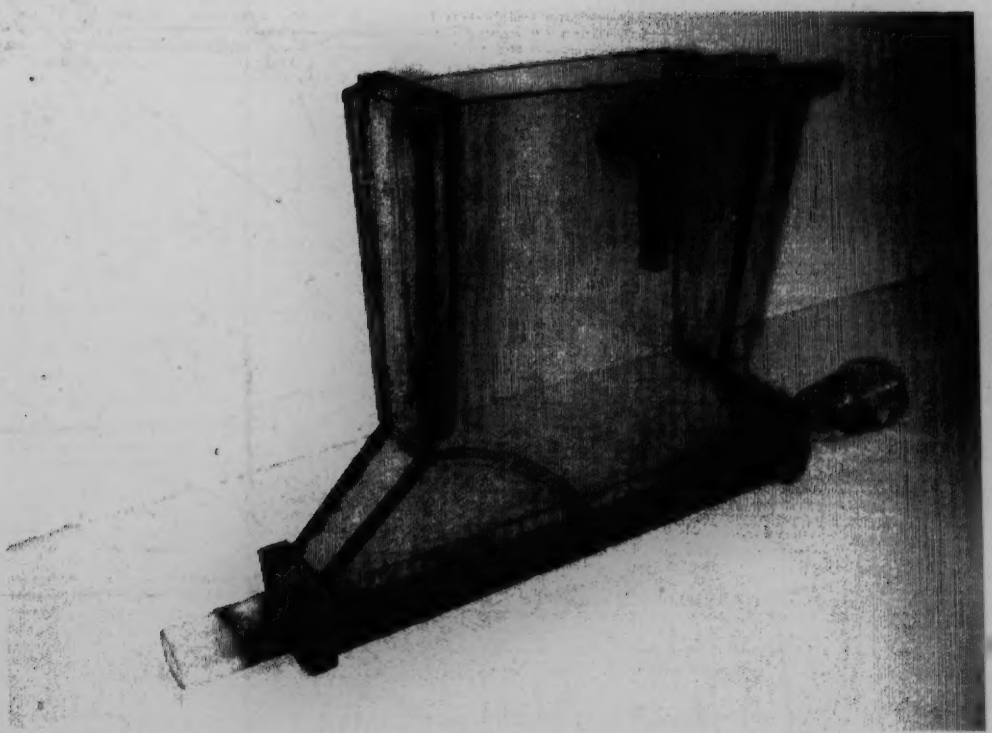

Figure A-12 Loading/flotation vessel with integrated cell for bubble visualisation

APPENDIX B: TABULATED DATA

Table B-1 Figure 3-1 Data

Figure 3-1	Time (minutes)			
Temperature	2	5	10	15
55°C	76.7	80.4	80.4	86.1
45°C	44.5	69.8	78.3	91.8
35°C	38.9	46.5	50.7	62.6
25°C	20.3	26.7	32.9	51.6

Table B-2 Figure 5-2 Data

Figure 5-2							
Test #1		Test #2		Test #3		Test #4	
Time	%	Time	%	Time	%	Time	%
2	24.4	2	27.5	3	34.0	1	14.7
5	44.4	5	44.0	6	48.7	2	30.1
15	67.8	15	64.3	17	71.0	5	45.9
						12	64.0
		32	78.5	30	79.5	30	77.9
60	84.3	60	84.6	60	83.8	60	83.1

Table B-3 Figure 5-2 Test Conditions

Test	1 - 4	Units
Process water source	de-ionised	
Process water pH	7.0	
Process water conductivity	0	mS
Process water bicarbonate	0	ppm
Process water chloride	0	ppm
Process water sulphate	0	ppm
Process water calcium	0	ppm
Ore source	Syncrude North Mine	
Ore type	Facies 11 Estuarine	
Bitumen content	12.3	%
Fines content (-44 µm fraction)	22.1	%
Sample size	1000	g
Slurry pH	8.3	
Pipe velocity	2	m/s
Air addition rate	150	sccm
Temperature	50	°C

1550 g for test #3

Table B-4 Figure 5-3 Data

Figure 5-3				
Time	Low 50°C	High 50°C	Low 35°C	High 35°C
0	0.0	0.0	0.0	0.0
1	68.8	13.3	8.8	0.5
2	87.1	24.8	17.9	1.2
3	93.1	32.5	30.2	1.9
4	95.7	39.8	41.4	2.9
5	97.0	45.0	52.1	4.4
6	97.6	50.7	61.7	5.9
7	98.0	54.2	70.7	7.7
8	98.3	58.4	77.2	9.9
9	98.5	60.6	82.3	12.9
10	98.6	63.3	86.5	15.8
11	98.8	65.9	89.9	18.8
12	98.8	68.1	92.2	21.8
13	98.9	70.3	93.8	24.8
14	98.9	72.9	94.9	28.8
15	98.9	74.6	95.6	32.5
16	99.1	76.7	96.3	35.3
17	99.1	77.9	96.8	38.5
18	99.1	79.6	97.2	42.2

19	99.1	81.0	97.4	45.0
20	99.2	82.4	97.8	48.9
21	99.0	83.3	98.0	52.2
22	99.0	84.5	98.2	55.7
23	98.9	85.5	98.3	57.8
24	99.0	86.6	98.4	61.2
25	99.0	87.6	98.5	63.6
26	99.0	88.7	98.6	65.7
27	99.1	89.2	98.7	66.9
28	99.1	90.1	98.8	70.0
29	99.1	90.9	98.8	71.9
30	99.1	91.5	98.9	74.2
31	99.1	92.3	98.9	75.5
32	99.2	92.7	99.0	77.7
33	99.1	93.5	99.0	79.0
34	99.2	93.8	99.1	80.3
35	99.2	94.1	99.1	81.9
36	99.2	95.0	99.1	83.6
37	99.2	95.7	99.2	84.4
38	99.2	96.2	99.2	85.2
39	99.3	96.3	99.2	86.5
40	99.2	96.7	99.2	87.8
41	99.3	97.2	99.2	88.4
42	99.3	97.4	99.3	89.4
43	99.3	97.6	99.3	90.3
44	99.2	98.0	99.3	91.0
45	99.3	98.2	99.3	91.6
46	99.3	98.5	99.3	92.4
47	99.3	98.6	99.3	93.1
48	99.4	98.8	99.3	93.5
49	99.3	98.9	99.4	93.9
50	99.3	99.0	99.4	94.5
51	99.3	99.1	99.4	94.9
52	99.3	99.2	99.4	95.4
53	99.4	99.3	99.4	95.7
54	99.4	99.3	99.4	96.0
55	99.4	99.4	99.4	96.3
56	99.4	99.4	99.5	96.7
57	99.4	99.4	99.4	97.0
58	99.4	99.5	99.5	97.2
59	99.4	99.5	99.5	97.3
60	99.5	99.5	99.5	97.6

Table B-5 Figure 5-3 Test Conditions

Test	Low Fines 50°C	Low Fines 35°C	High Fines 50°C	High Fines 35°C	Units
Process water source	Syncrude Base Plant	Syncrude Base Plant	Syncrude Base Plant	Syncrude Base Plant	
Process water pH	8.0	8.0	8.0	8.0	
Process water conductivity	4.07	4.07	4.07	4.07	mS
Process water bicarbonate	940	940	940	940	ppm
Process water chloride	750	750	750	750	ppm
Process water sulphate	210	210	210	210	ppm
Process water calcium	17	17	17	17	ppm
Ore source	Syncrude North Mine	Syncrude North Mine	Syncrude North Mine	Syncrude North Mine	
Ore type	Facies 11 Estuarine	Facies 11 Estuarine	Estuarine	Estuarine	
Bitumen content	14	14	9.5	9.5	%
Fines content (-44 µm)	15	15	30	30	%
Sample size	1000	1000	1000	1000	g
Slurry pH	N/A	N/A	N/A	N/A	
Pipe velocity	3	3	3	3	m/s
Air addition rate	0	0	0	0	sccm
Temperature	50	35	50	35	°C

Table B-6 Figure 5-4 Data

Figure 5-4							
Low Fines 50°C		High Fines 50°C		Low Fines 35°C		High Fines 35°C	
Time	%	Time	%	Time	%	Time	%
3	11.6	3	15.9	3	16.1	3	9.9
6	28.3	10	31.0	10	30.7	10	17.2
18	64.7	18	61.7	20	43.9	20	27.9
30	76.0	30	76.5	40	56.6	40	46.9
45	83.7	45	85.3	60	63.2	60	58.4
60	87.0	60	90.2				

Table B-7 Figure 5-4 Test Conditions

Test	Low Fines 50°C	Low Fines 35°C	High Fines 50°C	High Fines 35°C	Units
Process water source	Syncrude Base Plant	Syncrude Base Plant	Syncrude Base Plant	Syncrude Base Plant	
Process water pH	8.0	8.0	8.0	8.0	
Process water conductivity	4.07	4.07	4.07	4.07	mS
Process water bicarbonate	940	940	940	940	ppm
Process water chloride	750	750	750	750	ppm
Process water sulphate	210	210	210	210	ppm
Process water calcium	17	17	17	17	ppm
Ore source	Syncrude North Mine	Syncrude North Mine	Syncrude NW Quad	Syncrude NW Quad	
Ore type	Facies 11 Estuarine	Facies 11 Estuarine	Estuarine	Estuarine	
Bitumen content	14	14	9.5	9.5	%
Fines content (-44 µm)	15	15	30	30	%
Sample size	1000	1000	1000	1000	g
Slurry pH	N/A	N/A	N/A	N/A	
Pipe velocity	3	3	3	3	m/s
Air addition rate	200	200	200	200	sccm
Temperature	50	35	50	50	°C

Table B-8 Figure 5-5 Data

Figure 5-5		
Time (minutes)	Air:Slurry = 1:200	No Additional Air
3	16.1	15.7
10	30.7	28.1
20	43.9	35.2
40	56.6	40.1
60	63.2	41.9

Table B-9 Figure 5-5 Test Conditions

Test	1:200 air	No Air Addition	Units
Process water source	Syncrude Base Plant	Syncrude Base Plant	
Process water pH	8.0	8.0	
Process water conductivity	4.07	4.07	mS
Process water bicarbonate	940	940	ppm
Process water chloride	750	750	ppm
Process water sulphate	210	210	ppm
Process water calcium	17	17	ppm
Ore source	Syncrude North Mine	Syncrude North Mine	
Ore type	Facies 11 Estuarine	Facies 11 Estuarine	
Bitumen content	14	14	%
Fines content (-44 µm)	15	15	%
Sample size	1000	1000	g
Slurry pH	N/A	N/A	
Pipe velocity	3	3	m/s
Air addition rate	200	0	sccm
Temperature	35	35	°C

Table B-10 Figure 5-6 Data

Figure 5-6				
Time (minutes)	1:87	1:200	1:900	No Air
3	8.7	9.9	8.1	7.2
10	17.3	17.2	12.6	9.0
20	29.6	27.9	20.0	11.4
40	49.2	46.9	34.9	14.4
60	61.4	58.4	45.6	17.4

Table B-11 Figure 5-7 Data

Figure 5-3				
Time	Low 50°C	High 50°C	Low 35°C	High 35°C
0	0.0	0.0	0.0	0.0
1	0.5	1.8	0.7	0.7
2	1.2	4.3	1.9	1.7

3	1.9	7.3	3.3	3.2
4	2.9	10.6	5.1	5.4
5	4.4	14.1	7.3	8.4
6	5.9	17.9	10.2	11.5
7	7.7	22.1	12.9	15.5
8	9.9	25.8	16.4	19.2
9	12.9	29.5	20.4	24.3
10	15.8	32.8	23.9	29.1
11	18.8	36.8	28.2	33.8
12	21.8	40.4	31.6	40.1
13	24.8	44.0	35.7	45.3
14	28.8	46.8	40.1	50.5
15	32.5	51.0	45.1	55.2
16	35.3	54.1	49.3	60.2
17	38.5	57.2	53.1	64.1
18	42.2	61.4	56.8	67.5
19	45.0	63.5	61.2	70.3
20	48.9	66.7	64.2	73.6
21	52.2	69.0	68.9	77.8
22	55.7	71.8	71.9	80.8
23	57.8	73.6	75.0	83.7
24	61.2	75.6	77.4	85.9
25	63.6	77.9	80.5	88.1
26	65.7	79.5	82.9	89.9
27	66.9	81.0	84.7	91.4
28	70.0	82.8	86.2	92.5
29	71.9	84.2	87.7	93.7
30	74.2	85.5	89.3	94.8
31	75.5	86.5	90.6	95.5
32	77.7	87.5	91.8	96.2
33	79.0	88.5	92.6	96.7
34	80.3	89.7	93.4	97.1
35	81.9	90.6	94.2	97.5
36	83.6	91.3	94.8	97.7
37	84.4	91.7	95.3	98.0
38	85.2	92.5	96.0	98.2
39	86.5	93.1	96.4	98.4
40	87.8	93.5	96.7	98.6
41	88.4	94.1	97.0	98.8
42	89.4	94.5	97.3	98.9
43	90.3	94.9	97.7	98.9
44	91.0	95.3	97.9	99.0
45	91.6	95.6	98.0	99.1
46	92.4	95.7	98.2	99.2
47	93.1	96.1	98.4	99.2

48	93.5	96.3	98.5	99.3
49	93.9	96.5	98.6	99.3
50	94.5	96.8	98.6	99.4
51	94.9	97.0	98.8	99.4
52	95.4	97.1	98.8	99.4
53	95.7	97.4	98.9	99.4
54	96.0	97.5	99.0	99.5
55	96.3	97.7	99.1	99.5
56	96.7	97.8	99.1	99.5
57	97.0	97.9	99.2	99.5
58	97.2	98.0	99.2	99.6
59	97.3	98.1	99.2	99.6
60	97.6	98.2	99.3	99.6

Table B-12 Figure 5-6 and 5-7 Test Conditions

Test	1:87	1:200	1:900	No Air	Units
Process water source	Syncrude Base Plant	Syncrude Base Plant	Syncrude Base Plant	Syncrude Base Plant	
Process water pH	8.0	8.0	8.0	8.0	
Process water conductivity	4.07	4.07	4.07	4.07	mS
Process water bicarbonate	940	940	940	940	ppm
Process water chloride	750	750	750	750	ppm
Process water sulphate	210	210	210	210	ppm
Process water calcium	17	17	17	17	ppm
Ore source	Syncrude North Mine	Syncrude North Mine	Syncrude North Mine	Syncrude North Mine	
Ore type	Facies 11 Estuarine	Facies 11 Estuarine	Facies 11 Estuarine	Facies 11 Estuarine	
Bitumen content	9.5	9.5	9.5	9.5	%
Fines content (-44 µm)	30	30	30	30	%
Sample size	1000	1000	1000	1000	g
Slurry pH	N/A	N/A	N/A	N/A	
Pipe velocity	3	3	3	3	m/s
Air addition rate	500	200	50	0	sccm
Temperature	35	35	35	35	°C

Table B-13 Figure 5-8 Data

Figure 5-8			
Time (minutes)	1:200	1:900	No Air
3	12.7	9.2	0.0
10	46.5	26.8	4.1
20	68.5	42.7	4.1
40	84.0	61.7	16.3
60	90.0	66.2	18.3

Table B-14 Figure 5-8 Test Conditions

Test	1:900	1:200	No Air	Units
Process water source	Syncrude Base Plant	Syncrude Base Plant	Syncrude Base Plant	
Process water pH	8.0	8.0	8.0	
Process water conductivity	4.07	4.07	4.07	mS
Process water bicarbonate	940	940	940	ppm
Process water chloride	750	750	750	ppm
Process water sulphate	210	210	210	ppm
Process water calcium	17	17	17	ppm
Ore source	Syncrude Aurora Mine	Syncrude Aurora Mine	Syncrude Aurora Mine	
Ore type	Transition	Transition	Transition	
Bitumen content	8.8	8.8	8.8	%
Fines content (-44 µm)	33	33	33	%
Sample size	1000	1000	1000	g
Slurry pH	N/A	N/A	N/A	
Pipe velocity	3	3	3	m/s
Air addition rate	50	200	0	sccm
Temperature	35	35	35	°C

Table B-15 Figure 5-9 Data

Figure 5-9			
Time (minutes)	air:slurry = 1:900	air:slurry = 1:200	No additional air
3	0.0	0.0	0.0
10	0.0	0.0	0.4
20	0.8	1.3	1.4
40	2.6	3.8	6.7
60	5.8	9.3	11.5
80	24.0	32.9	19.7

Table B-16 Figure 5-9 Test Conditions

Test	1:900	1:200	No Air	Units
Process water source	Syncrude Base Plant	Syncrude Base Plant	Syncrude Base Plant	
Process water pH	8.0	8.0	8.0	
Process water conductivity	4.07	4.07	4.07	mS
Process water bicarbonate	940	940	940	ppm
Process water chloride	750	750	750	ppm
Process water sulphate	210	210	210	ppm
Process water calcium	17	17	17	ppm
Ore source	Suncor Steepbank Mine	Suncor Steepbank Mine	Suncor Steepbank Mine	
Ore type	Oxidised	Oxidised	Oxidised	
Bitumen content	11	11	11	%
Fines content (-44 µm)	13	13	13	%
Sample size	1000	1000	1000	g
Slurry pH	N/A	N/A	N/A	
Pipe velocity	3	3	3	m/s
Air addition rate	50	200	0	sccm
Temperature	35	35	35	°C

Table B-17 Figure 6-2 Data

Figure 6-2							
0.3 ml/min		0.15 ml/min		No kerosene		No kerosene repeat	
Time	%	Time	%	Time	%	Time	%
15	4.3	15	5.0	20	1.3	15	0.7
25	13.4	30	18.2			30	2.4
40	34.7	45	41.4	40	3.8	45	5.1
60	78.0	60	67.7	60	9.3	60	8.5
80	101.2	80	98.4	80	15.2	80	12.8

Table B-18 Figure 6-3 Data

0.15 ml/min kerosene		no kerosene	
Time	%	Time	%
7	8.7	10	3.2
15	18.9	20	6.8
30	36.2	30	10.6
45	51.0	40	14.1
60	66.4	60	19.8

Table B-19 Figure 6-4 Data

Time	Oxidised ore with no kerosene	Oxidised ore with 0.3 ml/min kerosene	Poor ore with no kerosene
0	0.0	0.0	0.0
1	7.3	9.4	5.7
2	12.8	11.3	10.4
3	16.5	13.5	13.7
4	19.7	15.7	16.4
5	22.6	18.1	18.6
6	24.8	20.3	21.1
7	26.6	23.2	22.7
8	28.8	25.4	24.9
9	31.0	27.8	26.7
10	33.3	30.1	27.5
11	35.3	32.4	29.3
12	37.4	34.2	30.9
13	39.2	36.3	32.8
14	40.9	38.1	34.1
15	42.5	40.3	35.1
16	44.5	41.9	36.2
17	46.4	43.9	37.5

18	48.0	45.5	39.3
19	49.8	46.8	41.0
20	51.6	48.4	41.9
21	53.8	49.9	43.2
22	55.2	50.6	44.9
23	56.6	51.5	46.2
24	58.3	53.0	47.3
25	59.8	54.7	48.7
26	61.4	55.8	49.9
27	62.5	57.3	50.6
28	63.3	59.0	52.2
29	64.3	59.9	53.4
30	65.9	61.6	54.2
31	67.3	62.7	55.6
32	68.5	64.0	57.0
33	69.6	64.9	57.9
34	70.8	65.8	59.5
35	72.0	67.2	60.5
36	73.0	68.4	61.6
37	74.4	69.0	62.5
38	74.9	70.1	64.1
39	75.8	71.0	63.2
40	77.1	72.2	65.1
41	77.8	72.9	66.7
42	79.0	73.8	68.1
43	80.1	75.1	68.7
44	81.1	75.9	70.0
45	81.4	76.5	70.7
46	82.5	77.7	72.1
47	83.5	78.2	73.0
48	84.3	78.8	73.7
49	83.8	80.6	74.7
50	84.4	81.4	76.0
51	85.1	82.2	76.9
52	86.4	83.0	77.7
53	87.0	83.3	78.2
54	88.3	83.6	79.8
55	88.1	85.0	80.3
56	89.3	86.0	80.8
57	90.1	87.2	82.2
58	89.9	87.9	82.7
59	90.9	88.0	83.1
60	91.9	88.5	83.4

APPENDIX C: SLURRY VISUALISATION SOFTWARE

C1 Visual Basic Form Code

The following is the computer code for the Pixelcounter.exe program used to analyze slurry images to determine information regarding the liberation of bitumen. This program is for use with a Euresys Picolo image capture board and calls several functions included with the Euresys drivers.

```
Option Explicit

Dim Buffer() As Byte ' The image buffer
Dim BufferSize As Long ' The size of the above
buffer (image width * height)
Dim frameCount As Long ' Counter for calculating
FPS
Dim totalFrameCount As Long ' Counter for total #
of frames
Dim writtenFrames As Long ' Counter for frames
written to file

Dim fileIsOpen As Boolean ' True if the data file
is open for appending

Dim pulse As Boolean

' Import PixelCounter2 function from the DLL
Private Declare Sub CountPixels2 Lib
"ImageProcessor.dll" _
    (bufPtr As Any, ByVal size As Long, ByVal lower
As Byte, ByVal upper As Byte, _
     ByRef below As Any, ByRef between As Any,
ByRef above As Any, ByRef average As Any)
```

```
Private Declare Function GetDigitalIOControl Lib
"ImageProcessor.dll" () As Long


' Import system function that tells time
Private Declare Sub GetLocalTime Lib "kernel32"
(ByRef ptr As Any)


Private Sub AutoFlash_Click()
    If (AutoFlash.value) Then
        Camera.SetExpParameterStr
eDigitalIoControl, "xxx0"
    Else
        Camera.SetExpParameterStr
eDigitalIoControl, "XXX1"
    End If
End Sub


' This will be automatically called whenever the
Board drop-down box is changed
' It fills the Source drop down box with
appropriate values for that board
Private Sub Board_Click()
    Channel.Clear
    Dim i As Integer
    For i = 0 To
EConfigPicolo1.SourceCount(Board.ItemData(Board.Lis
tIndex)) - 1
        Channel.AddItem i
        Channel.ItemData(i) = i
    Next
    Channel.ListIndex = 0
    Debug.Print "board changed"
End Sub


' Called when someone clicks on the browse button
(labeled "...")
```

```
' It pops up the dialog that asks for a data file
Private Sub BrowseButton_Click()
    CommonDialog1.Filter = "Comma Seperated Values
(*.csv)|*.csv|Text Files (*.txt)|*.txt|All Files
(*.*)|*.*"
    CommonDialog1.FileName = DataFile.Text
    'CommonDialog1.InitDir = DataFile.Text

    ' We don't want to overwrite the file name if
the user presses cancel, so we
    ' will tell it to generate an error on cancel.
If the error occurs, we will skip
    ' over the code that sets the file name in the
text box
    CommonDialog1.CancelError = True
    On Error GoTo SkipNameSet
    CommonDialog1.ShowOpen
    DataFile.Text = CommonDialog1.FileName
SkipNameSet:
    On Error GoTo 0 ' Turn off error handling

End Sub

' Called when the camera detects an error
Sub xCamera_Error(SurfaceHanlde As Long, ErrorCode
As enumECameraError, Data As Long, Flags As Long,
TimeCode As Long, Reserved As Long)
    If ErrorCode = eNoSignal Then
        MsgBox "No signal on channel " &
Channel.List(Channel.ListIndex) & "."
    Else
        MsgBox "Unknown camera error."
    End If
End Sub
```

```
' Called whenever the camera has a new surface
(image) available
Private Sub Camera_SurfaceAvailable(ByVal
SurfaceHandle As Long, ByVal Data As Long, ByVal
Flags As Long, ByVal TimeCode As Long, ByVal
ExtendedStatus As Long)
    frameCount = frameCount + 1
    totalFrameCount = totalFrameCount + 1


    ' Count the pixels
    If (DoConversion.value = 1) Then
        Dim below As Long
        Dim between As Long
        Dim above As Long
        Dim average As Long
        CountPixels2 Buffer(0), BufferSize,
LowerThreshold.Text, UpperThreshold.Text, below,
between, above, average
        BelowTextBox1.Text = below
        BetweenTextBox1.Text = between
        AboveTextBox1.Text = above
        AverageTextBox1.Text = average
        If (fileIsOpen And DoWrite.value = 1) Then
            Print #1, GetTimeStr() & "," & below &
"," & between & "," & above & "," & average
            writtenFrames = writtenFrames + 1
        End If
    Else
        BelowTextBox1.Text = ""
        BetweenTextBox1.Text = ""
        AboveTextBox1.Text = ""
        AverageTextBox1.Text = ""
    End If
    EBW8Image1.Refresh
```

```
    Camera.SetExpParameterStr eDigitalIoConfig,
"xxx1"
End Sub


Private Sub Flash_Click()
    Camera.SetExpParameterStr eDigitalIoConfig,
"xxx1"
    Camera.SetExpParameterStr eDigitalIoConfig,
"xxx0"
    Camera.SetExpParameterStr eDigitalIoConfig,
"xxx1"
End Sub

' Called once when the program first starts up
Private Sub Form_Load()
    DataFile.Text = Environ("USERPROFILE") & "\My
Documents\PixelCounterData.csv"
    Board.Clear
    Dim i As Integer
    For i = 0 To EConfigPicolo1.BoardCount - 1
        Board.AddItem "#" & i
        Board.ItemData(i) = i
    Next
    Board.ListIndex = 0 ' Board #0
    Board_Click ' Trigger filling of Source drop-
down
    Channel.ListIndex = 1 ' Composite BNC
    fileIsOpen = False
    PrepareCamera
    SetEnabled True
    Timer1_Timer ' Set the status line text
    AutoFlash_Click
End Sub
```

```
' Prepare the camera.  This allocates the image
buffer and makes the
' necessare setup calls.
Private Sub PrepareCamera()
    Dim SurfHandle1 As Long

    Camera.BoardId = "#" &
Board.ItemData(Board.ListIndex)
    Camera.VideoSource =
Channel.ItemData(Channel.ListIndex)
    Camera.ImageSizeX = ImageWidth.Text
    Camera.ImageSizeY = ImageHeight.Text
    BufferSize = Camera.ImageSizeX *
Camera.ImageSizeY
    ReDim Buffer(0 To BufferSize - 1) As Byte

    EBW8Image1.SetSize Camera.ImageSizeX,
Camera.ImageSizeY
    ' Sometimes we may want to use the simpler
memory allocation method
    ' for debugging.
    If (True) Then
        EBW8Image1.SetImagePointer
VarPtr(Buffer(0))
        SurfHandle1 =
Camera.CreateNewSurface(VarPtr(Buffer(0)),
Camera.ImageSizeX, Camera.ImageSizeY,
Camera.ImageSizeX)
    Else
        SurfHandle1 =
Camera.CreateVisionSurface(EBW8Image1.Object)
    End If
    Camera.AddSurface SurfHandle1
End Sub

' Check if the board or source has changed, and if
so re-prepare the camera
```

```
Private Sub CheckCamera()
    If (Camera.VideoSource <>
Channel.ItemData(Channel.ListIndex) _
        Or Camera.BoardId <>
Board.List(Board.ListIndex)) Then
        PrepareCamera
    End If
End Sub

' Called when the Grab button is clicked
Private Sub Grab1_Click()
    SetEnabled False
    CheckCamera
    Camera.Grab
    SetEnabled True
    Grab1.SetFocus
End Sub

' Called when the Live button is clicked
Private Sub Live1_Click()
    SetEnabled False
    CheckCamera
    frameCount = 0
    totalFrameCount = 0
    writtenFrames = 0
    Open DataFile.Text For Append As #1
    fileIsOpen = True
    Camera.Live
    Timer1.Enabled = True
    Timer1_Timer ' Set the status line text
    Stop1.SetFocus
End Sub
```

```
' Called when the Stop button is clicked
Private Sub Stop1_Click()
    Timer1.Enabled = False
    Camera.Stop
    fileIsOpen = False
    Timer1_Timer ' Set the status line text
    Close #1
    SetEnabled True
    Live1.SetFocus
End Sub

' A helper function to enable/disable most of the
text boxes
Private Sub SetEnabled(value As Boolean)
    Stop1.Enabled = Not value
    Grab1.Enabled = value
    Live1.Enabled = value
    Board.Enabled = value
    Channel.Enabled = value
    ImageWidth.Enabled = value
    ImageHeight.Enabled = value
    LowerThreshold.Enabled = value
    UpperThreshold.Enabled = value
    DataFile.Enabled = value
    BrowseButton.Enabled = value
End Sub

' Convert the local time from GetLocalTime to a
human-readable String
' Format: yyy/mm/dd hh:mm:ss.000
Private Function GetTimeStr() As String
    Dim LocalTime(0 To 7) As Integer
    GetLocalTime LocalTime(0)
```

```
    GetTimeStr = _
        LocalTime(0) & "/" & _
        LocalTime(1) & "/" & _
        LocalTime(3) & " " & _
        LocalTime(4) & ":" & _
        LocalTime(5) & ":" & _
        LocalTime(6) & "." & _
        LocalTime(7)
End Function


' This is called whenever the Timer1 interval
occurs.
' It will update the FPS indicator on the status
bar
Private Sub Timer1_Timer()
    If (fileIsOpen) Then
        Status.Caption = totalFrameCount & " Total
Frames.  " _
        & writtenFrames & " Frames Written.  " _
        & (frameCount / Timer1.Interval) * 1000 & "
FPS."
    Else
        Status.Caption = totalFrameCount & " Total
Frames.  " _
        & writtenFrames & " Frames Written.
Stopped."
    End If
    frameCount = 0
End Sub
```

C2 Image processor code

The following is the code for the MS C++ Imageprocessor.c program called by the pixelcounter.exe program to count the pixels in the retrieved slurry image.

```
#include <windows.h>
#include <stdio.h>
#include <sys/timeb.h>
#include <time.h>

UINT __stdcall CountPixels(LPVOID buf, UINT size,
BYTE thresh)
{
     UINT below = 0;
     unsigned char * start = (unsigned char*)buf;
     unsigned char * end = start + size;
     for ( ; start < end; start++)
     {
          if ( *start < thresh ) below++;
     }
     return below;
}

void __stdcall CountPixels2(
     LPVOID buf,
     UINT size,
     BYTE lower,
     BYTE upper,
     LPVOID belowPtr,
     LPVOID betweenPtr,
     LPVOID abovePtr,
     LPVOID average)
{
     UINT below = 0;
     UINT above = 0;
     unsigned long sum = 0;
     unsigned char* current = (unsigned
char*)buf;
     unsigned char* end = current + size;
     while (current < end)
     {
          if ( *current < lower ) below++;
          if ( *current > upper ) above++;
```

```
            sum = sum + *current;
            current++;
        }
        *((UINT*)belowPtr) = below;
        *((UINT*)betweenPtr) = size - above - below;
        *((UINT*)abovePtr) = above;
        *((UINT*)average) = sum / size;
    }

    UINT  __stdcall GetDigitalIOControl()
    {
        return '\n'; // "xxx0\nxxx1";
    }
```

C3 Pixelcounter program form

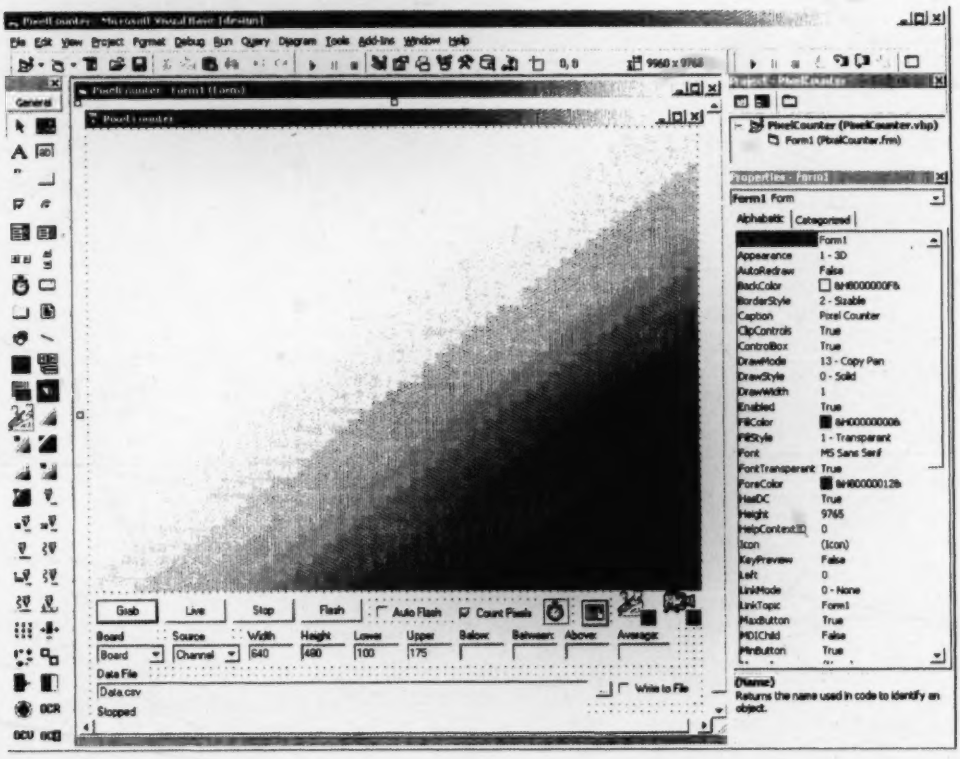

Figure C-1 Screenshot of the Pixelcounter program Visual Basic form

APPENDIX D: LHES MATERIALS LIST

Supplier	Quantity	Product Code	Description
Pegasus Glass 1-800-315-0387	2	R6905-016	PTFE 1" expansion Joint
	2	PS 1/100	Conical Glass Flange Ends
	2	ACF 1	1" ACF Flange
	4	CN 1	1" ACF Flange Insert
	2	TMEG	1" ACF Teflon Gasket
Edmonton Valve and Fitting 437-0640	2	SS-16-UT-1-16	Ultra Torr Fitting 1"
Order Phoned In	1	SS-65TF16	Valve, 1" Female NPT
	1	SS-65TS16	Valve, 1" Swagelok ends
	1	SS-1610-3TFT	1" Tee
	2	SS-1611-PC	Port Connectors
	1	SS-12-HC-1-16	Hose Connectors
Bock Optronics 1-800-762-1570	1	1155	Picolo Capture Board
	1	TM-7EX	B&W CCD Camera
Contact: Hans Bock	1	PVCS-2.0-C	Cable
	1	AO8080	Spot Lens
	1	AO8410	Dual spot gooseneck
	1	PD-12	Power Supply
	1	MVS-2601	Strobe Source
	1	88-0199	MVS-20 Adaptor for Fostec Bundle
Asian Computers 780-437-9504	1		Motherboard & Chip
	1		Ram Upgrade
Contact: Dan Bilsborrow	1		HDD Upgrade to 30 GB
	3		ATA 100 30 GB HDD
	1		Promise IDE Raid Controler
	1		Additional IDE Cables
	1		3rd Cooling Fan
	1		250 MB Zip
	1		CD-RW
	1		Matrox G450
	1		Optical Mouse
	1		Monitor Samsung 950P
	1		Soundblaster live value
	1		Basic Speakers
	1		Ulead Video Studio Pro
Fisher Scientific 1-800-2fisher	4	09-656H	Whatman 43/123 Thimbles
PO# 30-725668 Phoned in on Contract Release	1	09-873K	Whatman 934-AH Filter Papers
Ref# 725668	1	10-205D	250 ml Volumetric Flasks (Case 12)
ANIK Ext. 531	1	11-473-68	Flask Heater
Placed Feb 5/2001	1	13-873-5	External Temperature Probe
	4	11-471-5B	Glas-Col Heating Mantle w/o control
ACCT# 507001	1	13-874-7	Circulating heater/chiller
UofA Technical Resource Group	1		50' ethernet cable
UofA CNS	1		Office 2000
	1		Windows 2000 pro
	1		Visitech

APPENDIX E: DEAN STARK METHOD

Preparation

1. Dry Thimbles, Centrifuge Tubes, and Filter Papers in Vacuum oven for 1 hour at 120 degrees C. Store dried materials in dissector until needed.

Procedure

1. Place Thimble in 8 oz. Jar and weigh to nearest 0.1 mg.
2. Transfer Sample to Thimble.
3. Place Thimble in Extractor, record weight of empty 8 oz jar.
4. Add 250 ml of Toluene to extractor.
5. Reflux the extractor without overflowing the thimble for 3 hours or until all water is collected in trap and toluene dripping from thimble is colourless.
6. Add 3 drops of Isopropyl Alcohol in top of condenser to bring down any water.
7. Allow extractor to cool, remove water from trap to pre-weighed container and weigh.
8. Disassemble extractor, placing thimble in original 8oz. Jar and dry overnight in 120 C vacuum oven.
9. Remove bitumen/solvent solution to 250 ml volumetric flask, rinsing with toluene and dilute to volume after cool.
10. Invert flask several times to suspend fine solids and then transfer 30 ml to a clean pre-weighed dry centrifuge tube. Centrifuge at 15,000 RPM (21,000 G) for 15 minutes.
11. Place 5ml aliquots of centrifuge supernatant on pre-weighed and numbered filter papers and dry for 19 minutes. After allotted time weigh filter + bitumen. (note drying time must be calibrated for each fume hood used)

12. Rinse solids remaining in centrifuge tube with toluene until clean being careful not to dislodge solids. Dry tubes in 120 C vacuum oven for 1 hour and let cool in desiccator then weigh.
13. Drain water from trap into pre-weighted container being careful to shut off valve when toluene/water interface reaches tip of trap. Weigh container and subtract tare to get water content of sample.
14. Calculate the bitumen content of the sample by subtracting the bitumen-laden filter paper from the tare and multiplying by 250ml then dividing by 5ml.
15. Calculate the Solids content of the sample by taking the mass of the centrifuge tube plus dried solids and subtracting the tube tare, multiply the resulting solids mass by 250 and divide by 30, this gives the mass of the fines in the hydrocarbon phase. Add the mass of the fines in the hydrocarbon phase to the difference between the thimble plus dried solids and the thimble tare to get the total solids in the sample.

APPENDIX F: SLURRY VISUALISATION CALCULATION DETAILS

Data derived from the visualisation program is scanned for outliers and these outliers are removed from the data set before it is condensed. An outllier is classified as any data point that is more than two standard deviations away from the average of the previous 10 measurements (excluding previously identified outliers). The standard deviation is calculated from the same previous 10 valid data points. From time to time the strobe that illuminates the image fails to fire and results in significantly outlying data that needs to be removed or it would skew the results.

After the data set is purged of outliers it needs to be condensed. The image analysis program analyses 15 images per second, resulting in approximately 54,000 data points in a one hour test. This is too many to plot so the data is averaged into one-minute intervals.